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ASSESSMENT OF CERAMIC-MATRIX COMPOSITE TECHNOLOGY AND POTENTIAL DoD APPLICATION

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John E. Hove
H. Mauzee Davis

December 1977

Prepared for

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INSTITUTE FOR DEFENSE ANALYSES
SCIENCE AND TECHNOLOGY DIVISION

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At the request of the Assistant Director for Engineering Technology, ODDR&E, IDA conducted a study of ceramic-matrix composite (CMC) technology for the purpose of assessing (1) the potential usefulness of such materials to DoD systems, (2) the present scientific and engineering state of affairs for such materials, (3) whether the DoD should contemplate new projects for such materials, and, if so, (4) what should be the technical nature and direction of such projects.

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Approximately nine man-months of direct IDA effort were devoted to this task. The methodology included a survey of the technical opinions of about 300 knowledgeable scientists and engineers, and a workshop of about 15 members of the scientific community who were actively involved in CMC technology. The purpose of these was not to establish any type of consensus, but rather to establish up-to-date technology base and opinion data which the authors then assessed in order to form their own critical judgments. It should be emphasized that there are very few ongoing DoD projects or applications for CMCs; this study, therefore, is almost entirely concerned with possible future plans.

Areas of beneficial applications are outlined, and it appears clear that the potential usefulness of CMCs to DoD systems is high and very broad in scope. Although there are a few exceptions, it does not seem to be quite so clear which areas of the technology are likely to prove the most promising for instigating future programs. Nonetheless, the knowledge base in existence today is substantially greater than that of just a few years ago, and the authors applied their best judgment in attempting to formulate suggested initial projects for DoD consideration.

Two options, as frameworks for possible future actions, are offered. Option I contains what the authors consider to be the highest priority program, at an estimated level of effort of 16 man-years the first year; Option II contains additional somewhat lower priority but highly desirable projects which add up to an estimated total of about 29 man-years the first year. Since these are all new and speculative projects (with varying degrees of technical risk), no attempt is made to formulate the program for the second year and beyond; this must be influenced by the results of the R&D. If the DoD feels that the application benefits are sufficiently desirable, the general recommendation is made that one of these options (or some variation thereof) be adopted and implemented.

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ASSESSMENT OF CERAMIC-MATRIX COMPOSITE TECHNOLOGY AND POTENTIAL DoD APPLICATION

John E. Hove
H. Mauzee Davis

December 1977

INSTITUTE FOR DEFENSE ANALYSES
SCIENCE AND TECHNOLOGY DIVISION
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Acknowledgment is made of the highly competent and cooperative role of the respondents to the IDA survey on ceramic-matrix composites, and of the workshop participants. These persons are listed in the appendices of this report. In addition, the authors wish to express their deep appreciation to those persons who supplied them with critical comments after reviewing an early draft of the text. The reviewers are Dr. Maurice Sinnott of the University of Michigan, Dr. Richard Spriggs of Lehigh University, and Drs. Ernest Bauer and Donald Dix of IDA. Their comments were gratefully received and most useful.

ABSTRACT

At the request of the Assistant Director for Engineering Technology, ODDR&E, IDA conducted a study of ceramic-matrix composite (CMC) technology for the purpose of assessing (1) the potential usefulness of such materials to DoD systems, (2) the present scientific and engineering state of affairs for such materials, (3) whether the DoD should contemplate new projects for such materials, and, if so, (4) what should be the technical nature and direction of such projects. Approximately nine man-months of direct IDA effort were devoted to this task. The methodology included a survey of the technical opinions of about 300 knowledgeable scientists and engineers, and a workshop of about 15 members of the scientific community who were actively involved in CMC technology. The purpose of these was not to establish any type of consensus, but rather to establish up-to-date technology base and opinion data which the authors then assessed in order to form their own critical judgments. It should be emphasized that there are very few *ongoing* DoD projects or applications for CMCs; this study, therefore, is almost entirely concerned with possible future plans.

Areas of beneficial applications are outlined, and it appears clear that the potential usefulness of CMCs to DoD systems is high and very broad in scope. Although there are a few exceptions, it does not seem to be quite so clear which areas of the technology are likely to prove the most promising for instigating future programs. Nonetheless, the knowledge base in existence today is substantially greater than that of just a few years ago, and the authors applied their best judgment in attempting to formulate suggested initial projects for DoD consideration.

Two options, as frameworks for possible future actions, are offered. Option I contains what the authors consider to be the highest priority program, at an estimated level of effort of 16 man-years the first year; Option II contains additional somewhat lower priority but highly desirable projects which add up to an estimated total of about 29 man-years the first year. Since these are all new and speculative projects (with varying degrees of technical risk), no attempt is made to formulate the program for the second year and beyond; this must be influenced by the results of the R&D. If the DoD feels that the application benefits are sufficiently desirable, the general recommendation is made that one of these options (or some variation thereof) be adopted and implemented.

CONTENTS

EXECUTIVE SUMMARY

A. INTRODUCTION

At the request of the Assistant Director for Engineering Technology, ODDR&E, IDA conducted a study of ceramic-matrix composite (CMC) technology for the purpose of assessing (1) the potential usefulness of such materials to DoD systems, (2) the present scientific and engineering state-of-affairs for such materials, (3) whether the DoD should contemplate new projects for such materials in its future Materials and Structures Technology Base programs, and, if so, (4) what should be the technical nature and direction of such projects. Approximately nine man-months were devoted to this task, including IDA personnel and consultants. Dr. H.M. Davis, coauthor of this paper, is a consultant to IDA.

A ceramic-matrix composite is defined as a material with a continuous homogeneous ceramic phase, reinforced with a second (or more than one) phase, which may or may not be continuous. The second phase(s) may be either particulate or fibrous and may be either ceramic or metallic. The major purpose of any CMC is to attempt to overcome the single largest disadvantage of ceramics, their extreme brittleness, while continuing to take advantage of their excellent high-temperature properties, their corrosion resistance, and their hardness. In the present study, only structural applications were considered where the component had to withstand tensile stresses with a high reliability.

B. METHODOLOGY

The major approach to accomplish the objectives of this study was to utilize, as much as possible, the expertise of the entire U.S. technical ceramics community. First, a technical survey questionnaire (Appendix C) was sent to over 300 knowledgeable persons in the science and engineering of high-performance ceramics. Second, an intensive workshop was attended by 15 persons selected for their present interest and experience in CMCs. Third, was the meeting of a five-man panel, which had access to both the survey and the workshop, to attempt to summarize the key points brought out. Also, the authors conducted a literature search and held numerous private discussions.

It should be emphasized that the end purpose of gathering this vast amount of technical information from such a wide source was not to seek a *consensus* of opinions. In fact, it is probably next to impossible to obtain a meaningful consensus on such a complex subject as evidenced by the summaries of the opinions expressed in the survey and in the workshop (Chapters III and IV of the text). Rather, the end purpose was to obtain a reasonably complete and up-to-date data base of technological status and a broad spectrum of informed opinion from which the authors could draw conclusions based on their assessment of the information. Thus, the final recommendations represent the considered judgment of the authors. Since it is impossible to justify such judgments on any rigorous basis, it is recognized that other observers might draw a somewhat different set of conclusions. For this reason, as much of the raw data and opinions as feasible is included in the text.

C. FINAL ASSESSMENTS AND RECOMMENDATIONS

The final assessments and recommendations of the study are summarized below in the same sequence as the list of objectives given earlier. It should be emphasized that this is not a matter of assessing the efficacy of what the DoD is presently doing;

essentially, there is no ongoing DoD program of sufficient size to warrant a critique. Rather, then, what is being done is to assess the future potential of CMCs, starting from practically a zero present applications base.

1. Potential Usefulness of CMCs to DoD Systems

a. Short-Term (<5 years) Applications

Seeker Domes. Many of the present radomes are made of slip cast fused silica which presents problems in both integrity and rain erosion. There has been past work on quartz filament-wound/silica materials which showed promise.

Reentry Vehicle (RV) Antenna Windows. Present quartz/silica or boron nitride/boron nitride are marginal performers as pointed out in an earlier IDA paper (Ref. 1). DoD work on high-performance boron nitride fibers is almost at a standstill and should be reinvestigated for this as well as other applications.

Concrete Structures. Iron wire-reinforced concrete is almost reaching a commercial stage, particularly through the efforts of Battelle on selling their Wirerand® process. The DoD has been somewhat remiss in not applying a larger effort to investigate the potential uses of this and similar reinforced concrete (or cement) composites.

Armor for Kinetic-Energy Penetrators. The analytical ability to predict how successful armor arrays should be built is presently very limited. A systematic experimental program to empirically outline the materials systems needed appears to be in order. One supposes, with some confidence, that reinforced ceramics (including laminates in this case) will have a high priority.

Moderately Increased Temperature Gas Turbines. A moderate increase of uncooled gas-turbine inlet temperatures to 2000°F (with excursions up to about 2200°F for short periods) may

be feasible using silicon carbide fiber-silicon composites. While obviously not as exciting as temperatures of 2500°F or more, such applications do offer the promise of increased fuel savings and perhaps at low cost (by comparison with superalloy materials).

Erosion-Resistant RV Nosetip. This is a seemingly pressing problem that has been covered in a previous IDA paper (Ref. 1). The emphasis here is that the tantalum carbide/graphite composite (see Appendix A, page A-15) and the boron carbide/graphite composite (see Appendix A, pages A-40 and A-42) are promising enough that they should be considered strongly for applied system efforts.

Shipboard Multifunctional Incinerator. An ongoing Navy 6.3 program, this device is having serious refractory liner problems due to the various criteria it must meet, not the least of which is being usable on board a ship (Chapter II, Section D-4). The Navy is presently attempting to use a thin castable refractory lining either sprayed or (more usually) troweled in place. It would appear feasible and advantageous to incorporate ceramic fibers (perhaps alumina or silicon carbide, depending on the precise matrix).

b. Long-Term (>5 years) Applications

Reinforced Glass Composites. Although not yet designed for a specific application, this type of material now appears to have sufficient basic data to indicate a high promise and to warrant a search for usefulness. The reader's attention is directed to Appendix A, Section 6. With a density close to carbon/polyimide (and much less than carbon/aluminum), carbon/glass has a potential use temperature of over 1200°F as opposed to 700-1000°F for carbon/aluminum. Although the present study has not looked hard at detailed applications, such investigations should be made.

Air-Breathing Engines. From a resources standpoint, the bulk of present DoD ceramics R&D lies in support of the ceramic

gas turbine, and almost all of this effort is concentrating on silicon nitride and silicon carbide. Most members of the ceramic community have expressed concern as to whether a monolithic ceramic can be successful (at least in taking full advantage of ceramic properties) for this application. This area engendered the largest number of suggestions where it was thought that CMCs ought to be researched.

Diesel engine designs are well along the way to using ceramics for their refractoriness and thermal insulation. However, further significant advances (particularly in areas of reduction of energy use) can evidently be made by making all-ceramic hot moving parts, thus taking advantage of the higher ceramic strength-to-weight ratios. To do this would probably require composites.

Preliminary designs for a closed-Brayton-cycle ship propulsion engine (Chapter II, Section D-2) indicate that if a ceramic heat exchanger could be built to operate for long periods (about 10,000 hours) at 2500°F, the specific weight of the power plant could be reduced by as much as a factor of ten. While a stationary device, the long-term thermal stresses and oxidation present extremely difficult problems. It may be necessary to have the components built of oxide materials for such long-term use. However, oxides would almost surely have to be toughened by compositing. This is a strong argument for concentrating some R&D effort on oxide-matrix composites, especially since it could also apply to short use-cycle-time engines.

c. <u>Other Applications</u>

There are a number of other less well-definable CMC applications. Two that should be mentioned here are ceramic bearings (most likely silicon nitride) and ceramic gun tube inserts (probably alumina or zirconia). Another longer term use might be nozzles for chemical laser systems.

2. The Present Technology for CMCs

Our understanding of the technology of most ceramic-matrix composite systems seems to be broadly increasing although still incomplete. There has been work (of varying degrees of scientific sophistication) on a large number of material combinations and further exploratory work searching for *new* chemical composite systems no longer seems justifiable. For those systems that have been investigated, however, greater knowledge is needed about the chemical and physical bonding characteristics between the matrix and the reinforcement. Almost all potential applications for CMCs involve high-temperature and a corrosive (particularly oxidative) environment, and often temperature cycling. Under these conditions, chemical reactions between the phases or with the external environment may have a deleterious effect on the performance of the component. In fact, examination of research results to date leads to the following general conclusion.

- Metal (especially refractory metal) fiber-reinforced ceramics are not promising for any high-temperature application in an oxidizing atmosphere. It does not matter whether the metal is grown *in situ* or not, and all attempts to protect the wire with oxidation-resistant coatings have failed.

For this reason, the authors have not recommended any further long-range R&D involving metal fibers. Proponents of metal-fiber/ceramic use point out that such composites have shown great increases in fracture energy and toughness (which is true) and that dense thick surface coatings of a homogeneous ceramic could overcome the oxidation problem. The authors are not impressed with the latter argument since even one propagating surface crack could cause oxidative failure.

Of the monolithic ceramic materials, silicon nitride and silicon carbide have had perhaps the heaviest R&D attention over

the past seven to eight years. They have very good oxidation resistance and "toughness" and are thus the prime candidates for air-breathing engine components and high-temperature heat exchangers. However, they are still somewhat of a compromise; their oxidation resistance is not as good as oxide materials and their inherent toughness does not approach that of metals. Successful attempts to increase the "toughness" were made several years ago by incorporating long silicon carbide fibers into a silicon nitride matrix. However, the only fibers then available were made by vapor depositing silicon carbide on a tungsten wire; such a fiber is limited (because of deleterious thermal degradation) to temperatures of use below about 1500°F and no more work has been done on this particular composite. Recently developed silicon carbide fibers, not using a metal substrate, give promise of removing this restriction. As far as oxidation resistance is concerned, it should be emphasized that the mechanisms and, particularly, the effect of long time at high temperature are imperfectly understood for silicon carbide and hardly known at all for silicon nitride. The authors are uneasy about the wide range of applications being promulgated for these materials without the existence of better corrosion data, especially at long times. In addition, possible "pest reaction" problems seem to be ignored.

Excellent technical results have been obtained in growing *in situ* oxide fibers and lamellae in oxide matrices by directional solidification (DS). The scientific problems seem well in hand. Several people, however, dismiss this technique (which is somewhat analogous to single crystal growth) as impractical since it is presently a long slow process. While possibly true, the opinion of the authors is that it may well be the *only* way in which useful fiber-reinforced oxides can be formed. Considering the potential importance of oxides with greatly increased "toughness," it would seem impractical *not* to focus some R&D attention on improving the fabrication techniques.

In general, CMC processing techniques form a very significant area for research attention. The use of classic methods for introducing brittle fibers (e.g., by mixing with the matrix powder and then hot-pressing) generally may not work well enough to ever warrant consideration as a manufacturing method; for one thing, the fibers often break up and become almost useless. As pointed out in the text (see, e.g., Chapter V, pages 61 and 62), several innovative processing methods of promise are in various stages of development for both fiber and particle composites. The authors consider this a vital area for longer range R&D programs.

Partially stabilized zirconia (PSZ) is a CMC with quenched-in metastable particles as a reinforcing phase. Introduced just a few years ago, it has excellent toughness and abrasion resistance and is becoming useful as an extrusion die in forming copper and brass wire. It is also true that no one seems to have a very complete explanation for just *why* it is so tough. While *not* standing in the way of application of PSZ, this lack of knowledge does inhibit the search for other ceramic systems which might exhibit similar behavior. Generalizing this problem, the mechanisms of toughening in composites are still not sufficiently clear, despite an impressive accumulation of knowledge. While no one would reasonably argue that the theory must be complete before applied studies are made, it does appear that increased R&D efforts would be fruitful in understanding the basic fracture mechanics of CMCs. Nondestructive evaluation also falls in this category.

Several CMC systems appeared to be sufficiently advanced, in the demonstration of both properties and processing, that they deserve to be discussed.

- **Fiber-Reinforced Concrete or Castable Refractory**

Most of the newer developments have been done under private auspices and have reached a stage for one system (chopped

iron wires in concrete) where large-scale demonstration efforts are under way.

- **Graphite Reinforced with Carbides**

The implacement of tantalum carbide fibers (formed *in situ*) in a graphite matrix appears to be at an advanced development stage. Under security wraps, this work has been under way in DOE facilities for many years and is believed to be a proven on-the-shelf material. Its usefulness is for very high temperatures in nonoxidizing environments. Particulate reinforcement by boron carbide in graphite is not as far along, but looks promising for reentry vehicle use.

- **Silicon Carbide-Carbon/Silicon**

There are various systems under development which have silicon as a matrix, reinforced with silicon carbide (or silicon-carbide-coated carbon) fibers or particles. Use temperature is limited to not much over 2000°F, but such materials appear to show good "toughness" characteristics with potentially inexpensive fabrication techniques. Mechanical property determination has not yet been extensive enough to warrant immediate exploitation, but many of these systems are quite well developed.

- **Nonmetal Fiber-Reinforced Glass**

Much R&D was done on CMCs using glass (including fused silica) matrices in the 1950s and early 1960s. There was then a hiatus until a year or two ago. For example, filament-wound silica fibers in a fused silica matrix was successfully developed for radome use, in about the mid-1960s, but then the technology appeared to drop from sight. Work has been done recently on introducing continuous fibers of carbon or silicon carbide into glass and subsequently forming complex structures. Both the strength *and* the toughness are improved with increasing fiber content, which appears to be a unique characteristic among CMCs. Use temperatures up to about 1200°F in an oxidizing

atmosphere now appear feasible. Although in early stages of development, the technological outlook seems very promising.

D. RECOMMENDATIONS FOR DoD ACTION

From the previous discussions, it would appear that the potential usefulness of CMCs to DoD systems is high and very broad in scope. With a few exceptions, it is not quite so clear which areas of technology are likely to prove the most promising for instigating R&D programs. Option I, below, presents one suggested program involving what appears to be the smallest and most effective investment of resources. Option II lists additional desirable projects if (or when) greater resources become available. CMCs, as a whole, form a cohesive technology with common basic problems regardless of the specific materials involved, and it is strongly recommended that the program be organized as a single coordinated DoD effort to permit the best use of funding.

OPTION I: SUGGESTED MINIMUM CMC PROGRAM

No attempt will be made to lay out a schedule or to delineate between in-house or contract work. Since almost all projects would be new starts (even if serving the needs of an ongoing program), only the first year's effort is indicated. It is understood that each project would be reviewed periodically with the direction or funding altered as appropriate. The suggested projects are listed in order of closeness to applicability and seeming potential. Estimated resources required for the first year, in man-years (MY), are shown for each. These estimates reflect a minimum meaningful project size based on the background experience of the authors.

1. <u>Near-Term (<2 years)</u>

1.1 <u>Ceramic Fiber/Castable Refractory</u>. To investigate chopped alumina fiber in typical high alumina refractory liners for use in newly developing Navy shipboard multifunctional incinerators. (3 MY)

2. Moderate-Term (2-5 years)

2.1 Nonmetallic Continuous Fiber/Glass. To investigate boron, carbon, and silicon carbide fibers imbedded in various glass matrices. An intensive applications tradeoff study is suggested as part of this. An existing Air Force project is about one man-year per-year-effort on alumina fibers for possible radome use. (2 MY)

2.2 Erosion-Resistant RV Nosetip. To investigate further existing carbide/graphite composites developed by Los Alamos and by NRL for RV nosetip application. There is a small existing Navy contract, estimated at about one man-year per year. (2 MY)

2.3 Oxidation Resistance of SiC and Si_3N_4. To investigate the stated problem so that reliable projections can be made to 10,000 hours. Also, the question of short-time low-temperature (about 900-1000°C) should be resolved. While not strictly a CMC project, the results could have a strong impact on the urgency of future CMC investigations. (1 MY)

3. Long-Term (>5 years)

3.1 SiC and Si_3N_4 Fibers/Si_3N_4. To investigate the use of the newer SiC long fibers in a Si_3N_4 matrix. In a concurrent part of the project, investigate the *in situ* incorporation of fibrous or whisker-like Si_3N_4 in the same matrix. (2 MY)

3.2 Oxide Fibers/Oxide. To investigate further *in situ* directionally solidified (DS) oxide growth in oxide matrices with special attention to improved processing techniques. This is an important backup technology in case SiC and Si_3N_4 prove impractical. (2 MY)

3.3 Sol-Gel Techniques for Carbides and Nitrides. To explore innovative sol-gel methods for precipitating (or coprecipitating) very fine particle composites, particularly relating to Sic and Si_3N_4, The Navy has some sol-gel in-house research. (1 MY)

3.4 *In Situ* Copyrolysis of Fibrous Precursors. To explore innovative methods whereby reinforcing fibers can be formed in place (within the matrix) by pyrolytic techniques. The Navy has a small in-house effort. (1 MY)

3.5 Toughening and Strengthening Concepts and NDE. To better understand the micromechanics of CMCs and potential non-destructive evaluation (NDE) techniques. The Navy has an in-house effort. (2 MY)

The total estimated resource effort required for Option I is 16 man-years for the first year.

OPTION II: SUGGESTED DESIRABLE CMC PROGRAMS

This option includes Option I and the following suggested projects.

1. Near-Term (<2 years)

1.1 Metal Fiber/Concrete. To investigate and expand on existing iron or steel-fiber-reinforced concrete for possible application to castable boat hulls. (2 MY the first year)

2. Moderate-Term (2-5 years)

2.1 Silicon Carbide/Silicon/Carbon. To investigate the application of existing technology of either preform SiC/Si or molded SiC/Si/C materials for medium-temperature gas turbine engines. (2 MY)

2.2 CMCs for Gun Barrel Liners. To investigate the various concepts and their potential benefits whereby CMCs could be applied to the gun barrel erosion and wear problems. (1 MY the first year)

2.3 CMCs for Ceramic Bearings. To investigate concepts whereby ceramic roller and ball bearings (especially Si_3N_4) might have their life and reliability significantly increased by the use of CMCs. (1 MY the first year)

2.4 Boron Nitride Fibers/Boron Nitride. To investigate techniques of producing high-performance boron nitride (BN) fibers and incorporating them into a dense BN matrix. The first application would be RV antenna windows, but there will be others depending on the results. (2 MY)

2.5 Alumina, Silica Fibers/Alumina, Silica. To reinvestigate the fabrication of filament-wound reinforcement in a silica or alumina matrix for erosion-resistant radome use. The fibers could be quartz or alumina. This could be a 6.3A project. (2 MY)

3. Long-Term (>5 years)

3.1 CMC Fabrication Techniques. To investigate the utility of innovative CMC fabrication methods. This would include a more thorough look at flame spraying, plasma spraying, chemical vapor deposition, explosive forming, etc. Expertise exists in the country for each of these methods (albeit for other uses). (3 MY)

The supplemental estimated required resources are 13 man-years, so that the total resources for Option II are 29 man-years for the first year.

If the DoD decides that the military applications described above are needed, then the recommendation is made that the DoD consider and implement one of the two suggested options.

I. TASK DEFINITION AND APPROACH

In December 1976, the Assistant Director for Engineering Technology, ODDR&E, requested IDA to conduct a study of ceramic-matrix composite (CMC) technology for the purpose of assessing (1) the potential usefulness of such materials to DoD systems, (2) the present scientific and engineering state of affairs for such materials, (3) whether the DoD should contemplate new projects for such materials in its future Materials and Structures Technology Base programs, and, if so, (4) what should be the technical nature and direction of such projects.

Approximately nine man-months were devoted to this task, including IDA personnel and consultants. The scope of the effort was limited to materials meant for structural applications where the component had to withstand applied thermomechanical stresses without deleterious damage. There are, of course, many cases where the usefulness of the material is also limited by other than its mechanical properties. An obvious example is the seeker dome for missile radar or infrared acquisition and guidance systems where the material must have the appropriate electromagnetic properties. Such requirements, however, form an initial screening process for allowable materials; following this, in order to be useful, one must be able to choose and fabricate candidate materials that will also withstand the structural environment. It is this latter problem which is considered in the present paper.

It is appropriate to define what is meant by a ceramic-matrix composite. As used herein, the term refers to a material with a continuous homogeneous ceramic phase, reinforced with a

second (or more than one) phase that may or may not be continuous. The second phase(s) may be either particulate or fibrous and may be either ceramic or metallic. Some semantic confusion could arise, if the second phase is both metallic and continuous (e.g., continuous metal wires), as to what constitutes the "matrix" and what constitutes the "second phase." This question is usually resolvable by the relative amount of the phases and will not normally constitute a problem; however, it may on occasion, especially in the context of a "cermet." The term, cermet (a shorthand form of ceramic and metal), was coined sometime in about the 1950s to denote a material combination of a ceramic and a metal which would result in a marriage of the most desirable properties of each. Despite a great deal of R&D effort, the concept was rarely successful and usually resulted in a combination of the least desirable properties of each. Looking back on this work, from a present-day vantage point, it can be argued that the problem was less in the concept than in the unsophisticated approaches used to try to implement the concept. Nonetheless, there was at least one successful cermet application in the metal-bonded carbide cutting tool where a discontinuous majority phase (e.g., tungsten carbide particles) is bonded together by a continuous minority phase (e.g., a cobalt alloy). Such a material is not, by the above definition, a ceramic-matrix composite, but it is somewhat awkward to call it a metal-matrix composite as there is such a small amount of metal (perhaps 5-10 volume percent). This may be a unique case and the semantic problem will not be belabored any longer. It only indicates that the scope of the present study is necessarily somewhat arbitrary.

The class of carbon-carbon composites will not be considered herein. Although falling within our definition and of significant DoD importance, their military applications largely fall in the areas of reentry vehicles and rocket nozzles that have been analyzed and assessed in a previous IDA paper (Ref. 1). For

similar reasons, little will be said about erosion-resistant composites meant largely for nosetips of ballistic missile reentry vehicles.

Because of the relatively small amount of IDA effort associated with this task and because there is so little ongoing DoD R&D in the ceramic-matrix composite area, the major approach to accomplish the objectives of the study was to utilize, as much as possible, the expertise of the entire U.S. technical ceramics community.

First, questionnaires were sent to a selected list of over 300 individuals judged to be especially experienced in the metallurgy, physics, chemistry, and applications of high-performance ceramics. The questionnaire specifically requested the recipient's opinions as to applications, state of technology, and promising lines of future endeavor. About 100 useful replies were obtained, many of the rest responding that they had not considered the problem enough to indicate opinions. The useful responses represented a totality of about 1200 man-years of technical ceramics experience and enabled a good "first cut" at defining the problem.

The second step was to assemble a two-day workshop of a small number of specialists to explore ceramic-matrix composites in greater depth. This workshop was held at IDA on 8-9 September 1977 and was attended by about 15 individuals, chosen because of their personal involvement in ceramic-matrix composites. The representation included government, university, and industry. Because of the exploratory nature of the subject matter, participants were asked not to prepare formal papers, but to introduce their topic by a short talk and then lead the discussion. In order to summarize the results of the workshop, three of the attendees (plus the authors) convened a separate half-day meeting in a free discussion (which was taped) to try to summarize the information acquired. Although better in some respects than others, this technique of an intensive workshop plus an immediate

summarization worked well. The authors, of course, still had the job of condensing the ideas and approaches into a coherent recommended DoD preliminary program plan.

One weakness, by hindsight, of the workshop methodology used by the authors was the relative deemphasis of the materials/systems design interaction. This was purposely done because it was initially thought that the basic technology was sufficiently lacking to render such an applications effort unimportant in anything like detail. While this is still probably true for high-temperature uses, it does not seem to be true for some medium-temperature applications where detailed cost-benefit analyses should be carried out in the immediate future.

II. OVERVIEW OF TECHNOLOGY

A. CERAMIC STRUCTURAL MATERIALS

Before discussing the area of composites, it is worthwhile to review briefly, for the nonspecialized reader, the technology of ceramic materials, in general, and where it is of interest to DoD systems. Only structural applications are considered. More detailed background information may be found in Ref. 2.

A ceramic is usually defined as an inorganic refractory nonmetallic material in order to delineate it from the classes of polymeric plastics and of metal alloys, and from solid salts and other (inherently low-temperature) inorganic materials (such as sulfur). While the dividing line is frequently fuzzy (for example, graphite is the only elemental material considered to be a ceramic, but boron and silicon, both inorganic nonmetals, have respectably high melting points and their classification is certainly moot), this definition will serve for present purposes.

Some of the positive properties of selected ceramics that make them of interest for structural uses are discussed below.

1. Retention of Strength to High Temperatures

Most ceramics have essentially an identical yield strength and tensile strength; unlike most metals, the yield strength does not usually decrease very much until very high temperatures are reached and so the ceramic will usually maintain a high tensile strength to higher temperature than will a metal. Since many of the interesting structural ceramics have a low density (relative to high-temperature metal alloys), such materials often

show a marked superiority to metals in the high-temperature strength-to-weight ratio. Figure 1 illustrates this. The top figure shows typical values for some superalloys and refractory metal alloys (shown by trade names whose explanation is not relevant here, except to say they are representative of the commercially best available) while the bottom figure shows values for a few lightweight ceramics. The strength/weight ratio parameter is important in any structure where weight is a vital factor and, as seen in Fig. 1, the ceramics tend to show up better above about 800°C (1472°F). Ceramics also usually have a lower rate of creep (the tendency to distort with time at a constant applied stress) than metals at any given temperature.

2. Stiffness and Hardness

Most all ceramics have elastic moduli which are generally high by comparison with metals. Such stiffness is important in enhancing the dimensional stability and resistance to buckling of any structural member. From a dynamic stability standpoint, sensitivity to lower frequency modes of vibration is also reduced. Taken by itself, the importance of a high elastic modulus can be great. The reader may recall that a prime justification for the boron or graphite fiber-reinforced epoxy composites was the high stiffness-to-weight ratio obtainable. High hardness is a well-recognized property of ceramics and is exemplified in the widespread use of carbides and oxides for cutting and forming tools. From a structural standpoint, a major value of high hardness is the ability of the material to resist damage by an erosive environment such as might occur on a missile nose when traveling through rain.

3. Corrosion Resistance

As a general rule, ceramics can be selected (depending on the type of environment) that are much more resistant to corrosion, at high temperatures, than are metals. As an example, oxides (almost by definition) are fairly well immune to an

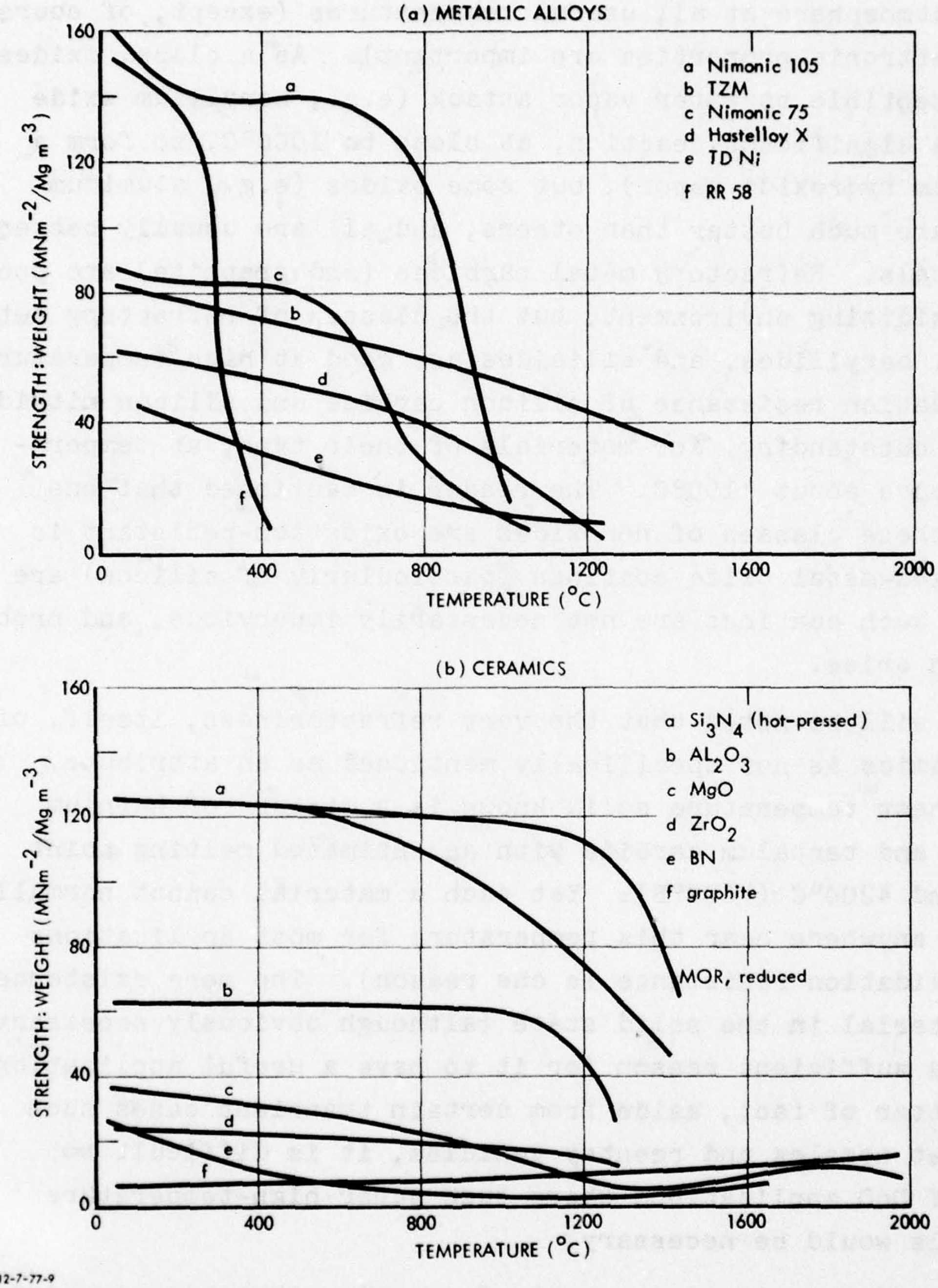

FIGURE 1. Variation of the Tensile Strength-to-Weight Ratio with Temperature for (a) Metallic Alloys, and (b) Ceramics (from Ref. 3)

oxygen atmosphere at all useful temperatures (except, of course, when electronic properties are important). As a class, oxides are susceptible to water vapor attack (e.g., beryllium oxide begins a significant reaction, at close to 1000°C, to form a beryllium hydroxide vapor), but some oxides (e.g., aluminum oxide) are much better than others, and all are usually better than metals. Refractory metal carbides (and graphite) are poor in an oxidizing environment, but the classes of refractory metal borides, beryllides, and silicides are good at high temperatures. The oxidation resistance of silicon carbide and silicon nitride is also outstanding, for materials of their type, at temperatures above about 1100°C. The reader is cautioned that one reason these classes of nonoxides are oxidation-resistant is that mixed-metal oxide coatings (particularly of silicon) are formed; such coatings are not necessarily impervious, and problems can arise.

It will be noted that the very refractoriness, itself, of the ceramics is not specifically mentioned as an attribute. The highest temperature solid known is a mixture of hafnium carbide and tantalum carbide with an estimated melting point of around 4200°C (7600°F). Yet such a material cannot normally be used anywhere near this temperature for most applications (poor oxidation resistance is one reason). The mere existence of a material in the solid state (although obviously necessary) is not a sufficient reason for it to have a useful application. As a matter of fact, aside from certain transient cases such as rocket nozzles and reentry vehicles, it is difficult to think of DoD applications where such super-high-temperature materials would be necessary.

Two of the negative aspect of ceramics that have impeded their more general structural use are given below.

1. **Brittleness**. Perhaps better defined as lack of toughness, this property is common to all ceramics. It is the inherent other-side-of-the-coin of the high-yield strength and

creep resistance. The strong, highly directional interatomic bonding and the complex crystal structure of ceramics simply do not permit enough of the short-time lattice plastic flow needed to blunt and stop crack growth under stress from being catastrophic. There is, of course, some resistance to crack propagation, one factor of which is frequently called fracture surface energy (this is something of a misnomer, but it is in common use to describe a parameter which includes the crack surface energy). An overall parameter frequently used to describe the tendency of a crack to increase in length under a given stress is called the stress intensity factor (usually labelled K_{IC}) which is proportional to $\sqrt{E\gamma}$ where E is the elastic modulus and γ is the fracture surface energy referred to above. The larger K_{IC} is, the better from a toughness standpoint. The K_{IC} of even the best (i.e., toughest) ceramic suitable for structural use is a factor of two or three lower than that of a brittle metal alloy. The value for typical refractory oxide would be a factor of ten lower. As a rule, all structural materials have built-in cracks and flaws, due to the fabrication process. For a given applied tensile stress (whether mechanical or thermal), it can be shown that the critical crack length (i.e., a crack which, if any longer, would propagate through the material and cause fracture) is proportional to the square of K_{IC}. It follows that for a structural component designed to withstand a given stress, cracks in a ceramic of the order of 10 to 100 times smaller than those in a metal must be detected by nondestructive testing before the component can be judged as safe to use. In absolute terms, this can mean that flaws of the order of 10 microns in size must be detected and classified, which is a difficult task for the existing technology of nondestructive evaluation. In addition, there is the problem that subcritical crack sizes can still grow under the proper conditions of thermal or mechanical cycling. This phenomenon is somewhat similar to fatigue in metals, although it is not completely understood.

In general, it can be stated that the difficulties of designing with inherently brittle materials is the most critical barrier to taking advantage of the otherwise desirable properties of ceramics.

2. Fabrication and Quality Control. With the exception of glassy materials, most ceramics are formed by mixing the powder (usually with a binder), pressing or extruding, calcining (which eliminates the binder) and finally firing at a high temperature such that the particles sinter together to make a monolithic body. Frequently, sintering aids (in the form of deliberate impurities) are added to the powder to obtain high densities. Another way of aiding the sintering is to start with very fine powders. Unless the chemical composition is such that a colloid-gel can be used, this usually means mechanical milling of the powder before mixing and pressing. Thus, it can be seen that there are difficult problems in maintaining uniformity, porosity, microstructure, and purity in a typical fabrication facility. Aside from this, unless the processor chemically forms his own powder starting material, there is the problem of determining purity of the vendor's powders. While this acceptance analysis can be, and usually is, done routinely for cation (metallic) impurities, it is not as easily done for many anion impurities. One of the authors has encountered a situation where two seemingly identical batches of beryllium oxide powder produced very different densities of the end-product under the same processing conditions. The apparent culprit turned out to be a difference in the amount of sulfate ion impurity which was not initially checked. One additional problem is that a large shrinkage occurs during firing of any ceramic formed by the above techniques. This is controllable provided every other aspect of the process (e.g., purity, temperatures, etc.) is very precisely controlled, which normally they are not, nor probably can they be.

One method used to alleviate some of the difficulties, particularly the shrinkage problem, is to press at high temperatures. This is costly but can result in a dense piece processed nearly to net shape, and the cost may be worth it. Hot-pressing certainly does not solve all the problems, but is probably the best technique in normal use. Nonetheless, in any high-temperature sintering process, it is difficult to avoid contamination and, what may be equally important, difficult to keep impurities from agglomerating and forming unwanted local phases. Aside from other effects, such inhomogeneities would generally have a different expansion than the bulk material, which could tend (on cooling down the specimen) to form internal flaws. These fabrication difficulties have not been particularly serious in the past, since by far the overwhelming bulk of usage of ceramics has been for nonstructurally critical applications. Improvement of processing and forming techniques must be an integral part of any planning to use ceramics as critical structural members.

B. TYPES OF CERAMIC STRUCTURAL APPLICATIONS

Continuing the background discussion of ceramics, it is worthwhile to indicate briefly the types of structural applications that are presently of interest to the DoD. These are listed below for the orientation of the reader and most are considered more deeply in Section II-D and in Chapter V specifically devoted to composite applications.

1. Internal combustion engines
   - Open-cycle gas turbine components
   - Diesel engine components
2. External combustion engines
   - Closed-Brayton-cycle heat exchangers
3. Rocket motor nozzle components
4. Ballistic reentry vehicle thermal protection system

5. Electromagnetic windows
   Radomes and infrared domes
   Reentry vehicle antennas
6. Magnetohydrodynamic generator components
7. Rolling and sliding high-speed bearings and seals
8. Battlefield and aircraft armor
9. Inserts for gun barrels
10. Dental and orthopaedic implants
11. Roads, runways, and structures (largely concrete)
12. Shipboard incinerators

The materials criteria for some of these applications vary depending on required conditions (engines, for example, cover a broad range of uses). There are applications of particular interest to other agencies than the DoD, which are not included above. Two examples are energy storage (e.g., flywheels) and controlled-fusion power-plant components.

C. CERAMIC-MATRIX COMPOSITES TECHNOLOGY BACKGROUND

In Section A, it was pointed out that the inherent brittleness of ceramics is a major stumbling block for their use in engineering applications where tensile stress must be accepted and where a highly reliable structural element must be achieved. It was also shown that, while in principle monolithic ceramic bodies could be built to overcome this problem, the requirements for flaw sizes were so stringent as to make them difficult to detect, let alone to achieve in fabrication. If, then, ceramics are to be used in such applications, it would appear that the designer must accept a probabilistic mode of failure; that is, even after a reasonable level of quality control, he will only know that "this piece has a 90% chance of withstanding 50,000 psi tensile stresses," not that "this piece will withstand 50,000 psi tensile stress." In many cases this will be satisfactory, but also in many it will not. It would be

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obviously desirable if some way could be devised to make ceramics more forgiving or, to put it another way, have greater "toughness."

The only presently known methods of doing this, approaching or achieving practicality, is to introduce cohesive elements or barriers to crack propagation, in the form of discrete particles or fibers, into the ceramic materials. In other words, one can form a ceramic-matrix composite. A well-known example is the use of steel reinforcing bars in concrete. Rebars partly prevent cracks from spreading, but they also take up tensile stresses when the concrete has cracked and allow the rest of the concrete structure to withstand compressive loading (for which it is well suited). Attempts to do this in more typical ceramic bodies have met with mixed success. More often than not, an increased toughness was accompanied by a decrease in tensile stress. This is indicated in Fig. 2, which shows the result of British work incorporating chopped fibers into various ceramic bodies. The chart on the left side shows that the fracture surface energy (a measure of toughness) increases modestly to tremendously as fibers are added, although for most matrices there is a maximum fiber volume which can be tolerated. However, the chart on the right side shows that the relative strength of most of the same materials decreases markedly. An exception appears to be aligned carbon-fiber-reinforced glass, which evidently continues to get both tougher and stronger as the fiber volume is increased. It should be mentioned that the tensile strength was only measured in the direction of alignment; presumably in the other directions, its strength would also decrease. Nonetheless, as will be brought out later, fiber-reinforced glasses appear to be a very interesting class of materials, whose applications may be relatively short-term, although the technological reasons for their good properties are not yet entirely clear.

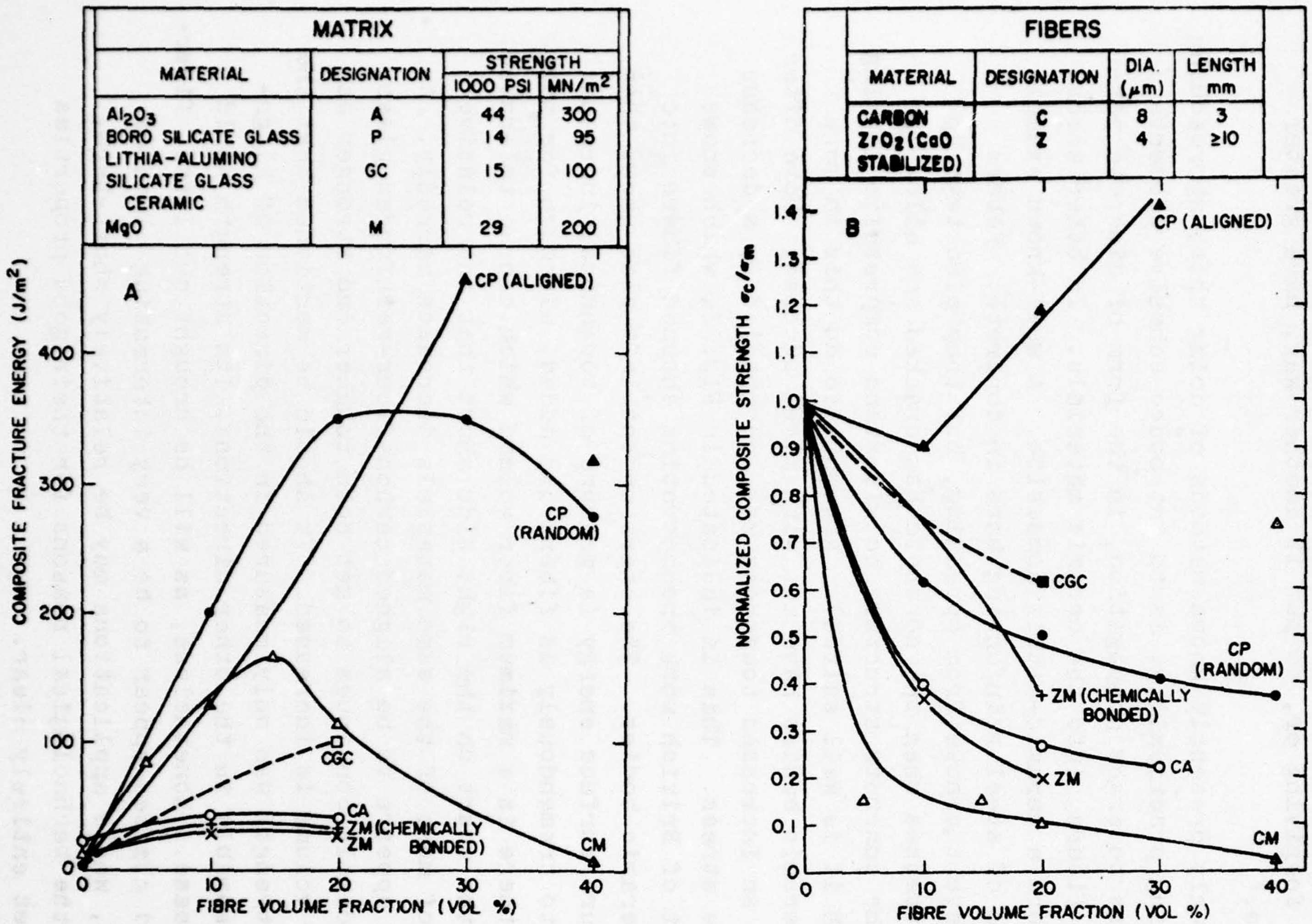

FIGURE 2. Some Measures of Toughness and Strength (at room temperature) for Various Fiber-Reinforced Ceramics (from R.L. Rice, IDA CMC Workshop; also, see Ref. 4)

Another illustration of the mixed blessings observed in early composite work is shown in Fig. 3. This shows the moduli of rupture (the breaking strength of a bar specimen under three-point loading) of aluminum oxide with various amounts of chopped molybdenum wires. The modulus is plotted against the number of thermal cycles from 2200°F to room temperature and it thus shows the effects of continued cracking tendencies on the strength, not just a single-temperature one-shot test. Note that pure alumina* essentially falls apart after two cycles, but the composite maintains a strength as far as the tests were carried out. However, similarly to the case of Fig. 2, the initial strength of the composite is appreciably lower than that of the matrix alone. This is at least partly due to the inadvertent increase in porosity as the fibers are introduced.

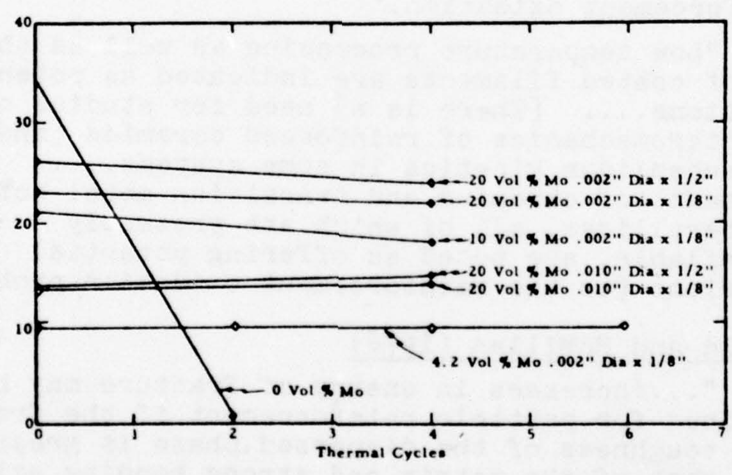

FIGURE 3. Modulus of Rupture as a Function of Thermal Cycling for the Alumina-Molybdenum Fiber System (Ref. 3)

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*Alumina is common shorthand for aluminum oxide, as is beryllia, magnesia, etc., for the oxide of the indicated metal.

The task of establishing the present technical state of affairs of ceramic-matrix composites was made easier by the existence of two excellent reviews: one was published by Krochmal (Ref. 5) about 10 years ago and the other by Donald and McMillan (Ref. 3) about one year ago. Both contain thorough references of work up to their times of appearance, and Krochmal gives a good compilation of phase equilibrium data. It is of interest to quote from each reviewer some of the more pertinent and technically broad conclusions.

Krochmal (1967)

"...despite a number of virtues of fiber reinforced ceramics, the present technology has been unable to capitalize on this approach to achieve more thermally shock resistant ceramics having predictable mechanical integrity. This [stems] from several factors, the most important being thermo-chemical interaction, cracking, and reinforcement oxidation.

"Low temperature processing as well as the use of coated filaments are indicated as potential solutions.... [There is a] need for studies of the micromechanics of reinforced ceramics [and of] subsolidus kinetics in some systems.... Filaments of chromium and transition metal borides and beryllides, all of which are presently unavailable, are noted as offering potential solutions [to the reinforcement oxidation problem]."

Donald and McMillan (1976)

"...increases in energy of fracture may be obtained for particle reinforcement if the fracture toughness of the dispersed phase is greater than that of the matrix and strong bonding exists, [and] crack deflection ...can marginally improve energy of fracture; however, in all instances, the increase is less than an order of magnitude and hence of questionable significance.

"Suitable fibre reinforcement can provide large increases in strength approaching an order of magnitude, and increases in energy of fracture may approach three or four orders of magnitude. [*Authors' note--evidently they refer here to glass matrices.*]

"Continuous brittle-fibre systems (i.e., carbon and silicon carbide fibres) have provided the most successful composites in terms of strength and toughness at ambient temperatures but they are of little practical value for extended elevated temperature use. [*Authors' note--this negative comment by D&M is due to the situation that the carbon filaments may readily oxidize above about 400°C, owing either to matrix porosity or cracking, and that the silicon carbide fibers prepared on a tungsten substrate will show thermal degradation above about 850°C. While both statements were true of the earlier work, recent developments in silicon carbide fiber technology and graphite fiber coatings indicate that these difficulties may be partially or totally overcome.*]

"For high fracture energy values with brittle fibres, pull-out effects are essential [*Authors' note--pull-out refers to the actual breaking of surface bonds and the physical pulling of fibers through part of the matrix as a crack spreads*], but rupture [or plastic deformation] of ductile fibres can lead to a high energy of fracture without the necessity for pull-out.

"...[chopped] brittle fibres yield only a small increment in energy of fracture whilst ductile fibers can provide large increments.

"...[there are uncertainties] of the in situ value of the fibre strength and, in the case of discontinuous reinforcement, ...of the effect of misaligned fibres as stress-concentration sites.

"Fibre reinforcement, particularly with ductile fibres, generally improves the resistance of a ceramic to thermal shock, but matrix microcracking may be initiated.

"The potential of fibre-reinforced ceramics is high, but only if several important aspects... are fully evaluated, can they be seriously considered as useful structural materials. [These aspects are:]

(i) dynamic and static fatigue resistance;
(ii) impact resistance;
(iii) thermal shock and thermal fatigue resistance;
(iv) long-term thermal stability;

(v) the effect of progressive matrix microcracking on these properties, and the effect of the environmental conditions and specimen size and shape."

As far as applications are concerned, Donald and McMillan have this comment:

> "At the present time [ca 1975-1976], fibre-reinforced glasses and ceramics have not provided viable alternatives to more conventional metallic materials, and much more work is required if such materials are ever to become acceptable replacements. However, their range of potential applications is extremely diverse and hence the incentive for further investigation is high."

Note the recurrence of statements to the effect that ductile (metallic) reinforcement is especially good in increasing the impact resistance and toughness of ceramic matrices, but that the oxidation problem greatly restricts the range of applications. Unfortunately, the latter problem is still with us. In 1966, Miller, Singleton and Wallace (Ref. 6) state:

> "The poor oxidation resistance exhibited by the composites casts doubt on the future of metal fiber reinforced ceramics at elevated temperatures particularly for use as jet engine components."

In 1976, Brennan (Ref. 7; also see Appendix A, Sections 1 and 5) states, in part:

> "One of the problems connected with the use of refractory metal wire...in a ceramic matrix composite...in an oxidizing environment [if a] crack develops [is that] catastrophic oxidation of the wire soon occurs."

Attempts were made to silicide the tantalum fibers used by Brennan. The coating was highly successful in preventing oxidation of the free wires, but had little effect in preventing their oxidation when incorporated into the ceramic composite (the reasons for this are not clear). For most of the high-temperature DOD application areas, there seems to be very little

encouragement for serious consideration of metal wire reinforced ceramics.

It is interesting to note that, even though ten years apart, both of the reviewers cited were in agreement that ceramic-matrix composites were not yet ready for use, at least in the more advanced structural applications, at the time their papers were written. By the time of Donald and McMillan (1976), the data information base had grown to the extent that they were able to delineate guidelines as to what the relative matrix and reinforcement properties should be. Since then the basic understanding has been further increased, especially by the work of the Naval Research Laboratory group (see Appendix A, Section 7), and there are now some areas where applications development efforts might bear fruit.

D. EXAMPLES OF POTENTIAL CMC APPLICATIONS

Section B above lists 12 potential application areas for ceramic structural use. It is worthwhile to briefly discuss a few of these from the viewpoint of ceramic-matrix composites. This is not intended to exhaust the list, but only to indicate some that appear to deserve fairly imminent attention.

1. Internal Combustion Engines

For reciprocating engines, the closest application is the diesel power plant as far as ceramics are concerned. R&D work, partially supported by the DoD, is in progress to exploit refractory, thermally insulating materials to such a degree that the conventional cooling system demands are either strongly alleviated or even totally eliminated. If this can be done, the operational advantages are obviously great. For example, it is presently estimated that almost 50% of the road maintenance problems associated with diesel-powered trucks are caused by the cooling system.

For open-cycle gas turbine use, of course, there are substantial (about \$3-5 million) efforts under way by DARPA to use silicon nitride and/or carbide materials as both stators and rotors. The program has had a limited success, but performance of test engines has been marginal because (among other things) of thermal stress, impact, and vibrational problems. The turbine inlet temperature has been downgraded to 2250°F (from 2500°F) for the Navy demonstrator engine. While there is a strong interest in the cruise missile application, no manned-aircraft uses are being contemplated, largely because of the inherently probabilistic mode of failure of the key ceramic parts.

For both these engine types, the ability to use an inherent "tough" ceramic would greatly enhance the probabilities of successful performance (i.e., performance where the full use of the ceramic advantages over metals can be utilized). As one specific example, rotor blade failures in the turbine demonstrator program have been ascribed (in a preliminary way) to vibrations considered excessive due to the very low internal damping characteristic of the ceramic. Compositing, in general, will add appreciable damping to such a material.

2. Closed-Brayton-Cycle Engines

Under ONR contract, feasibility studies are being carried out on compact lightweight closed-Brayton systems for ship propulsion. In such an engine, using an inert gas working fluid, the critical item from weight, volume, temperature, and corrosion standpoints is the heat source, and specifically the heat exchange system which sees the combustion products from a fossil fuel burner on one side and the inert gas on the other. One result of the study, done for a 40,000-hp engine, is that the specific weight of the power plant (in lb/hp) would be reduced by a factor of ten by using a ceramic heat exchanger operating with a hot-spot temperature of 2500°F. This would represent a quantum

jump in heat source technology, and make such engines look extremely attractive for shipboard use. Time between major overhauls for any such heat source would have to be several tens of thousands of hours. If the ceramic is silicon carbide or nitride, the apparent initial choices, there has to be a great concern about oxidation effects. Neither material has been tested for more than about 100 hours, and several people in the IDA workshop expressed concern about any long-term usage of these materials in an oxidizing atmosphere. At least some of the oxides would appear more satisfactory from this standpoint, but thermal stress problems then become more severe. An oxide matrix composite is an obvious choice, if within feasibility. Very likely, in order to preserve mechanical integrity, even silicon carbide would have to be reinforced.

3. Bearings

Ceramics, particularly silicon nitride, have made excellent roller and ball bearing materials, both with metal and with ceramic races. However, when they do break under load, they behave like typical ceramics and shatter catastrophically. If relatively inexpensive processing techniques can be developed for compositing such components, the feasibility of taking advantage of ceramic bearings might be enhanced.

4. Shipboard Incinerators

Multifunctional shipboard incinerators (MSI) would at first sight seem like mundane systems without significant problems. This does not appear to be the case.* An MSI combines the functions of a solid-waste incinerator, a freshwater sewage incinerator, and a saltwater sewage incinerator. It must be comparatively light and small (<10,000 pounds and to

*The authors are indebted to W.L. Wheatfall of DTSRDC, Annapolis, for supplying them with MSI information. MSI is an ongoing Navy 6.3 program. See also Ref. 8.

fit into a 12'x12'x8' space), have burn-time reliability for 10,000 hours between major overhauls and 1150 continuous hours without failure, withstand temperatures up to 2000°F in an extremely corrosive atmosphere, and withstand shock and vibration. If weight and size were not limiting, brute force refractory ceramic lining concepts could probably be used to satisfy the other requirements. Because of these limitations, however, the refractory liner is proving to be a significant R&D problem. This is a case where ceramic composite technology could be beneficially applied for a near-term problem.

III. SUMMARY OF RESPONSES TO THE IDA CERAMIC-MATRIX COMPOSITES SURVEY*

A. APPLICATIONS FOR CMC

The many suggested applications for ceramic-matrix composites (CMC), offered by the group of respondents,** were either founded upon (a) experience, (b) informed judgment, or (c) reasoned imagination. These potential uses may be classified, first, on an arbitrary basis of temperature of service; for example

1. near atmospheric temperature
2. about 100-700°C
3. above 700°C.

A second possible classification would divide the applications into those of potential value to the Department of Defense, and those of value to others. Because the needs of the DoD are so broad however, it does not appear fruitful to make that division at this time.

1. For Service Near Atmospheric Temperature

Suggested mechanical uses included roads, runways for aircraft (Duckworth, Hillig), tools and tooling (Baker, Diness, Richman), cutting tools (Whalen), and flywheels (Kupperman). As replacements for missing parts of the human anatomy, one notes the proposed use of CMC for prostheses (DeVries), and for dental and orthopaedic implants (Pollack). Brown suggested

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* Appendix C contains the questions used in the survey of the CMC Workshop.

** Respondents are listed in Appendix B in alphabetical order.

structural beams of wire-reinforced concrete. Beck thought of the potential virtue of CMC in hydrospace vehicles. Proposed applications with chemical overtones were as conveyor pipe for particulate or corrosive materials (Wirtz), as electrolyte in the sodium-sulfur battery (Stoddard), and as nozzles for chemical lasers (Stoddard).

Several envisioned uses of CMC were all akin because of their common relationship to electromagnetic radiation: window glass with metallic reinforcements (Chu, Brown, Ordway), antenna windows (Beasley, Diness), infrared transmitters of good strength (Wang), antimissile missiles (Richman), laser-proof aircraft (Baker), and laser-hard radomes (Bersch), or domes, in general (Beasley, Diness, Phillips, Rice, Steinberg). Tressler suggested rigidized fibrous insulation.

Suggestions for the battlefield were for armor (Baker, Cline, Davidson, Hillig, Kupperman), kinetic-energy penetrators or armor (Davidson, Richman), and antipersonnel fragmentation devices (Richman).

2. For Service About 100-700°C

Assignment of proposed applications to this roughly limited range of temperature is inevitably imprecise. Seemingly appropriate are bearings (DeVries), bearing surfaces (Herman, Krochmal), parts of piston engines (Hillig),* diesel components (Bortz), and Stirling engine heater-head tubes (Probst). High-temperature reinforced concrete (Wachtman) belongs here. Then come low-temperature burners (Doremus, Probst), flame arresters for rocket-boosted ramjets (Davidson), and environmental protection for coal-conversion reactors (Beasley). To these, one adds heat exchangers (Carpenter, Chaklader, Diness), high-

*In the mid-1960s, David Godfrey built a working lawn-mower engine with major components of silicon nitride. -HMD

temperature insulators (Davidson), aerospace vehicles (Beck), and high-temperature gas-cooled reactors (Chaklader).

3. For Service Above 700°C

Suggested applications for this demanding zone included containers for the processing of materials (Doremus), hot dies for refractory metals (Chapman), plate filters for combustion gas (Paquette), components for magnetohydrodynamic generators (Bradt, Chapman, Rice, Stoddard, Wachtman), furnace materials, furnace furniture, and crucibles (Doremus, Duckworth, McGee Phillips), and coal gasifiers, other high-temperature reactors (Wachtman), and gas-turbine combustors. Most often named were turbine components, their rotors, stators, and seals included (Bradt, Brennan, Bortz, Chaklader, Cline, Conrad, Diness, Doremus, Hillig, Krochmal, Lynch, McLean, Paquette, Prewo, Probst, Richman, Sato, van Reuth, Warren, Whalen, Wirtz). Rocket nozzles, and other hot nozzles, were also frequently cited (Ahmad, Carpenter, Cline, Davidson, Diness, Duckworth, Lynch, Wirtz). The potential value of CMC for aerospace was indicated by their suggested use in hypersonic vehicles (Baker, Krochmal), and in reentry surfaces; i.e., nose cones, leading edges, etc. (Beasley, Davidson, Duckworth, Kupperman, Lynch, Steinberg, Wirtz). Herman foresaw a probable role for CMC in the awaited technology of controlled thermonuclear reactions.

B. SUGGESTED MATERIALS COMBINATIONS

Respondents to the questionnaire suggested many combinations of materials which they thought worthy of investigation as possible ceramic-matrix composites. It is notable that the list contains remarkably few duplications, a fact that may well reflect a widespread uncertainty of knowledge of the field; until limitations are established through research, many of the systems offered in such a list will exhibit the influence of speculation.

Matrix	Reinforcement	Respondent
Al_2O_3	TiO_2	Herman
	ZrO_2	Herman, Phillips
	Steel	Ordway
	Tool Steels	Chaklader
	Stainless Steels	Chaklader
	Special Alloys	Chaklader
	Nickel	Chaklader
	Nichrome	Morgan, Ordway
	Mo	Ordway
	W	Ordway
	W by directional solidification	Ahmad
Aluminous Cement	Al_2O_3-SiO_2 Fibers	Tressler
Al_2O_3-Cr_2O_3	Cr	Stoddard
CaO	Mo	Stoddard
$LaCrO_3$	Cr and Pd	Stoddard
CeO_2	Mo	Chapman
Gd_2O_3	Mo	Chapman
SiO_2	SiO_2 Fibers	Pepper, Steinberg
	Al_2O_3 Fibers (duPont)	Steinberg
ThO_2	W-wire	Tinklepaugh
UO_2	W by Eutectic Solidification	Chapman, Wirtz
ZrO_2	ZrO_2 Fibers	Lynch
	Al_2O_3 Fibers	Lynch, Phillips
	BeO	Cline
	Nichrome	Machlin
	W by Eutectic Solidification	Chapman

ZrO_2-CeO_2-Y_2O_3		Stoddard
SiC	C-Fibers, Siliconized (Si Matrix)	Warren
	SiC (Si Matrix)	Whalen
	Si, Carburized	Burke, Probst
	BeO	Cline
	Pyrolytic Graphite	Graham
	Graphite	McLean
	SiC	Pepper
	Si_3N_4	McLean
SiC-Si_3N_4	Graphite Fibers	Pepper
HfC	ZrO_2	Davidson
TaC	Ni	Chaklader
	Mo	Chaklader
	Ti, etc.	Chaklader
TiC	Ni	Chaklader
	Mo	Chaklader
	Ti, etc.	Chaklader
WC	Ni	Chaklader
	Mo	Chaklader
	Ti, etc.	Chaklader
ZrC	ZrO_2	Davidson
Graphite	Nb-C	Hirada
	Ta-C	Hirada
AlN	ZrO_2	Cline
	Al_2O_3 and ZrO_2	Cline
	SiC Fibers	Graham

BN	Al_2O_3	Diness
	BN Fibers	Pepper
Si_3N_4	Refractory Metal	Pepper
	Ta Fibers	Ahmad, Bersch, Brennan, McLean, Machlin, Probst
	Ta or W wire	Tressler
	Fibers of SiC on W	Ahmad
	Superalloy Wires	Morgan
	Fibrous Si_3N_4	Diness, Pepper
Si_3N_4 "Reaction Bonded"		Burke, Richman
RSSN	Fibers made *in situ*	Graham
SiAlON	Al_2O_3 Fibers	Pepper
"Glass"	Al_2O_3	Lynch
	ZrO_2	Lynch
	Graphite Fibers	Bersch
	SiC Fibers	Brennan, Lynch
	Wires or Metal Fragments	Uher
	Fibers of High Elastic Modulus	Beck
Borosilicate	Mullite Fibers	McGee
Glass or Glass-Ceramic	Steel Fibers	Siefert
	B Fibers	Siefert

In the tabulation that follows, the distinction between matrix and reinforcement is usually firmly determined; but, occasionally, whenever the nature of the materials is such that one or both of them might well serve in either function, and the respondent has been less than clear in his presentation, the analyst's judgment has necessarily intruded, to that a measure of uncertainty exists.

Several respondents made general or incomplete specification of systems. Their contributions could not properly be fitted into the above tabulation, but are listed below so that their possible value may not be lost.

Partially Identified Systems	Respondent
Oxide Ceramic with SiC	McLean
Ceramic with Graphite	Baker
Glass-Matrix Composites	Prewo
Aluminum Silicate with Refractory Metal	Chu
Metal Carbide with Graphite	Davidson
Bonded Diamonds	DeVries
Porous Carbide infiltrated by Chemical Vapor Deposited (CVD) Metal	Davidson
Gels, Reinforced, Dehydrated	Mueller
Concrete with Glass Fibers	Blachere
Tough Stainless Steel in Castable Refractory	Smothers
Report: Feasibility of Fiber-Reinforced Ceramics	Milewski
Preview (1967) Giving Many Matrix-Fiber Combinations	Krochmal

C. GENERAL TECHNOLOGY

Many of the respondents offered cogent comments about the technology of ceramic-matrix composites. Although the remarks range widely in subject, they can be assigned, with at least passing satisfaction, to one of four categories: (1) Status of the Field, especially Processing; (2) Strengths, Advantages,

Promise; (3) Weaknesses, Disadvantages, Warnings; and (4) Suggested Improvements, Innovations.

1. <u>Status of CMC</u>

Alliegro reported that hot-pressed bauxite, reinforced with wire or mesh, served well in many situations involving wear or impact. Bradt thought that the processing of CMCs was a dominant concern. Chaklader referred to his research paper of October 1976 in which he told how to make a dense ceramic/metal body by hot-pressing particles of Al_2O_3 that had been coated with copper, then milled, then coated with nickel. Chu cited the importance of attention to six physical and chemical factors (composition, contact angle, interfacial adhesion, transformation, expansivity, and stress distribution), and to the principal variants of processing (e.g., atmosphere, diffusion, and rate of heating and cooling).

Cline disclosed his 1970 invention of honeycomb armor, a honeycomb of thin metal, filled with a ceramic powder, and the whole hot-pressed into a unitary structure. Pepper discussed (and Vasilos mentioned) a useful method of fabrication: a woven preform of the reinforcing fiber is impregnated with a slurry or a colloid containing the component for the ceramic matrix, and the body is then sintered or hot-pressed into the final shape. This procedure has been used for silica/silica and for BN/BN, but a source of inexpensive BN fibers is badly needed, as is a supply of Si_3N_4 fibers for a Si_3N_4/Si_3N_4 system. Phillips thought it feasible to add Al to ZrO_2, and Zr to Al_2O_3, to fire in hydrogen, then to fire in air to oxidize the metal, the final product being an oxide-reinforced oxide: Al_2O_3 in ZrO_2, and ZrO_2 in Al_2O_3. Pincus suggested that those who employ knitted or woven metallic wires might learn from the technology of glass-reinforced polymers. He pointed out that the use of a reinforcing metal in a ceramic required mastery of the principles

of combining unlike materials, for example, the relative expansivities and the relative elastic moduli. He wondered whether one might make use of clad metals, of principles that promote bonding, and of configurational preferences.

Russell, insisting that one should consider composites involving the more common ceramics--not only the rare and "exotic" materials, invited attention to ceramics of controlled porosity impregnated with plastics or metals, and to sintered ceramic-metal composites.

Tallan reasoned that the purpose of the second phase in a ceramic matrix should be to impart toughness or resistance to thermal shock, perhaps through crack arrest, crack branching, or other mode of absorbing energy.

Warren argued that, although the role of fracture surface energy in the mechanical behavior of brittle solids had received only minor direct R&D attention during the DARPA Turbine Program, improvement in the fracture energy of Si_3N_4 and SiC was nevertheless accomplished. This improvement was attributable to a reduction in impurities, and to a decrease in grain size through improved processing. Fibers of carbon or metal in a brittle matrix also increase the fracture surface energy (e.g., Mo fibers in Al_2O_3, Ta in Si_3N_4).

2. Strengths, Advantages, Promise

Bortz emphasized the potential advantages of fiber-strengthened ceramics, maintaining (a) that the fibers can carry the majority of the load, the matrix merely transferring imposed stress from fiber to fiber; (b) that fibers in a ceramic material can add toughness and lessen brittle fracture; (c) that, with low-density matrices, favorable strength-to-weight ratios are obtainable; (d) that an environment-resistant matrix will protect the load-bearing fibers; and (e) that, with appropriate design, the strength, fracture toughness, and brittleness of a CMC can be matched against requirements.

Diness saw promise of pullout toughening by fibers in a ceramic matrix, especially by fibrous Si_3N_4 in Si_3N_4. He also cited the potential for toughening through stress-induced transformation, and through the microworking that attends thermal cycling.

Both Fine and Firestone thought that castable refractories reinforced with fibers would offer enhanced strength and resistance to thermal shock.

Morgan pointed out that castable ceramics containing stainless steel fibers are in use in furnace roofs. He predicted that "graded" metal/ceramic structures, which could readily be made (e.g., by reactive hot-pressing), probably had an important future in turbines, etc.

3. <u>Weaknesses, Disadvantages, Warnings</u>

Alliegro considered composites "a risky business" beacuse, in service, reaction between phases, and interference because of expansion, would sometimes yield "the worst properties of the individual members." He therefore held that a composite ought to be used at temperatures that are well below the temperature of fabrication.

Bortz warned of possible incompatibility of the components of a composite. He explained the thermodynamic basis for predicting the compatibility or incompatibility of a paired metallic fiber and a ceramic oxide, and he supplied some relevant values of free energy of formation.

Cutler, writing of the potential virtues of glass fibers in the strengthening of building material, stated that the major problem was to discover an inexpensive matrix that would not degrade the fibers.

Duga said that a great impediment to progress in the field arose "from an incomplete knowledge of the characteristics of

the interfaces" in any ceramic/metal system of interest. He thought not only of the physical, chemical, mechanical, and structural characteristics, but also of how those interfaces were formed.

Lynch recognized many problems that make difficult the development of structural ceramic composites, especially chemical compatibility, differences in thermal expansion, and strength-to-weight ratios.

Paquette was impressed by the array of problems presented by the materials that are considered for use in CMC. Some of these were shear strength, toughness and impact strength, creep resistance, resistance to thermal shock, and long-term durability. He also saw the need for investigation of crack growth, fiber-matrix interaction, and susceptibility to corrosion/erosion/oxidation. He wondered whether the job was actually worth doing.

Pepper dwelt upon gaps in the technology, and upon the severity of the environment in which certain CMCs might be expected to serve. He considered the lack of suitable fiber reinforcement for high-temperature ceramics, such as silicon nitride, to be critical. He thought that, in particular, a fiber of silicon nitride exhibiting a high degree of chemical compatibility with bulk silicon nitride, and also possessing a matching thermal expansivity, was badly needed for the components of the gas turbine. He judged that the environment of heat exchangers for turbines and closed-cycle engines was equally demanding.

Like Alliegro, Searcy feared constitutional changes in CMCs during service, owing to changes in solubility limits with temperature. In thermal cycling, for example, components go in and out of solution. Even in a steady temperature gradient, the composition of a single phase may become inhomogeneous.

Tinklepaugh was dubious about the limitations of ceramic matrix/ceramic fiber composites "because they are still brittle."

Viechnicki, thinking of CMC grown *in situ*, concluded that a principal reason for the minimal activity in the area was that the gain in improved mechanical properties of such a system did not justify the difficult technology, that is, high temperature of melting, unsuitable crucible materials, loss by volatilization of the melt, great shrinkage with freezing, and faceted growth. Moreover, the product typically offers unexciting resistance to thermal shock.

In view of the current practice of industrial design, Warren looks upon the structural use of CMC as "a last-resort solution." High-cost, brittleness, poor reproducibility, flaw sensitivity, and paucity of design data have contributed to the reluctance of designers to consider ceramics, or CMC, for structural functions.

4. Suggested Improvements, Innovations

The many suggestions for the improvement of CMC are grouped under (a) General, or "Strategic," Comment; (b) Fabrication and Processing; (c) Special Materials; (d) Strengthening, Toughening.

a. General, or "Strategic," Comment. Bradt advised that one should be concerned with the fundamentals of structure/ property relationships. In similar vein, Wang maintained that only full attention to the topology of the component phases would bring composites into their proper place in engineering.

Courtney, to whom improved fracture toughness was the limiting need of ceramics, advised the study of (1) CMC with soft and/or ductile inclusions; (2) ceramic/metal *in situ* systems of either fibrous or lamellar morphology directionally solidified, and (3) ceramic/ceramic systems directionally solidified *in situ*. Hillig likewise recommended the attempt to achieve a desirable

reinforcement/microstructure through *in situ* reaction. Hillig also insisted that attention be given to the perfection, the stability, and the protection of the interphasal interface, and to the coupling between matrix and reinforcing phase(s). Hasselman thought that one could improve resistance to thermal stress by control of thermal conductivity.

To some respondents, the morphology of the composite was of surpassing importance. Urban suggested a sandwich of long, parallel fibers of Al_2O_3 between sheets of Al_2O_3, the interstices then being filled with a refractory metal or oxide. Steinberg, with his mind on the leading edge of the space shuttle, was inclined to put this trust in any sort of ablating or subliming structural system made up of a porous bonded-ceramic structure, impregnated with an organic material which when heated, would char gassily. McMillan thought that one would do well to imitate the structure of wood and bone.

b. Fabricating and Processing. A number of respondents proposed novel modes of fabrication. DeVries wondered whether a composite might be built up as a series of thin-film layers, possibly epitaxial. Graham, citing the reported preparation of equiaxed grain structures by chemical vapor deposition (manufacturers identified), concluded that CVD was a first-class candidate among methods for fabrication of CMC. Herman enthusiastically advocated the trial of plasma spraying for fabrication. Ordway suggested that a matrix (initially glassy for ease of fabrication) could be finished as crystalline by subsequent devitrification.

McGee proposed the addition of the matrix (presumably, to a reinforcement already in place) by the casting of a melt, by chemical infiltration, or even by CVD. With greater detail, Paquette endorsed fabrication by liquid or gaseous infiltration or impregnation, but he also saw probable economy in the preparation of ceramic bodies through pyrolysis of organic precursor

polymers. Probst et al. suggested burying fibers of Al_2O_3 in silicon, then nitriding to produce a reaction-sintered silicon nitride reinforced by alumina fibers.

Smyth averred that one could cast a fused ceramic refractory at, say, 2000°C, about reinforcing wires of a refractory metal, e.g., molybdenum. Wirtz recognized the prosaic cosintering of matrix and reinforcements, but he preferred to precipitate the reinforcing metal or ceramic from a ceramic matrix or, "more elegantly," to deposit both phases by eutectic solidification of an appropriate melt.

Confining his attention to ceramic/metal composites, Chaklader recommended coating the ceramic particles with metal before the unifying step of sintering or hot-pressing. He had found that energetic milling of the ceramic particles before fabrication was beneficial. He also thought that thermite reactions could be adapted to the deposition of the reinforcing metal, and to the fabrication of the composite body.

Quite alone in his assumption of the general viewpoint of management, Duga advised that, after development, the leap from laboratory to production must be attended by confidence in the testing methods employed for quality control. In other words, successful production must be guided by quality control which, in turn, rests upon reliable testing.

c. <u>Special Materials.</u> Wang thought that the effective infrared transmitter, Ag_3AsS_3, which is mechanically weak, might become valuable if built into a composite with a ceramic matrix which also exhibited passable IR transmission, e.g., MgO or Y_2O_3.

Cutler reported that he could produce inexpensive whiskers of SiC from clay, and that he would welcome the discovery of an appropriate matrix for them.

d. Strengthening, Toughening. Recognition of the need for enhanced toughness prompted several thoughful suggestions. Bradstreet conceived a CMC with a reinforcing phase of extremely high strength, and with a layer of ductile metal at the matrix-reinforcement interface for effective transfer of stress. Less definitively, he indicated conviction that there was virtue in crack arrest on a microscopic scale. Also writing with a broad pencil, McMillan declared that the road to toughness would pass through a thicket called "microstructural complexity." With more specific reasoning, Chapman maintained that aligned particles of a second phase offered promise as barriers to crack propagation, and McLean thought that preferred orientation of reinforcing fibers might be of paramount importance. A quite different approach was that of Cline and Rice, who suggested the purposeful addition of hard particles for initiating microcracks that would blunt potentially destructive cracks and thus deter their propagation.

The concept of providing toughness through the presence of a component that is subject to a strain-induced transformation was presented by both Diness and Hillig. Both were thinking of systems in which partially stabilized ZrO_2 was a disperse phase, subject to the tetragonal-to-monoclinic transformation when adequately strained.

Overlooking little, Kirchner suggested all three of the principal means of augmenting fracture toughness: introduction of small particles as barriers to crack propagation, enhancement of microcracking, and provision for strengthening by phase transformation.

Tressler proposed to strengthen by increasing the fracture energy, but he did not say how the improvement might be accomplished.

Duckworth summed up wide knowledge in few words. To inhibit initiation of fracture, ensure tight bonding at interfaces

between the matrix and a reinforcement of higher strength and elastic modulus. To inhibit propagation of cracks, ensure that the fracture surface energy will increase as cracking starts, e.g., by crack branching, by plastic flow, or by fiber pullout. The prevention of catastrophic failure by the incorporation of crack arresters, with attendant weakening of the ceramic matrix, requires that one accept the initiation of damage at a lower stress than that at which the unreinforced ceramic would remain intact.

D. NONTECHNICAL COMMENT

The comments of this section can appropriately be divided into (1) History, Administration, (2) Recommendations for R&D, and (3) Prospects.

1. History, Administration

Blachere was impressed that much of the early work on CMC, e.g., with metallic fibers, was apparently hasty, and that some of the promising results were forgotten. On the basis of long observation of CMC endeavor in Government, Bradstreet remarked that the prior effort was doomed by blunders of management, primarily stipulating that "available" materials be used, and failing to demand adequate information on the materials during the planning stage. The programs were further hampered by inadequate funding, by prejudicial pessimism in DoD and in NASA, and by a narrow view of the potential utility of CMC (i.e., only for refractoriness and wear resistance).

Thinking of limited expenditures for research in ceramics, Bersch expressed the opinion that R&D on CMC should be funded from the more generous provision for composites. Bradstreet thought that the planning of any "rational program" should be guided by a permanent advisory committee. Probst was convinced that one could not proceed far into a CMC program without

comparing the projected cost of the product with the predicted need, or market.

Diness reported that ONR was thinking of holding a workshop on toughening mechanisms in ceramics.

2. Recommendations for R&D

A strong proponent of basic research on CMC was Sato. Noting the lack of fundamental knowledge of such materials, he insisted that basic research was urgently needed, along with developmental research and the study of processing. In the same mood, Searcy declared that the chemical and microstructural effects of thermal cycling should receive "sophisticated and coordinated study." Likewise, Hillig urged that "emphasis should be on systems that make sense theoretically"; that materials work should be combined with theory, lest the effort be mainly productive of wishful thinking.

Both Doremus and Ordway were firm in the opinion that the best R&D program would proceed as an effort to solve an actual problem, rather than as a general development of materials; that one should identify specific applications, and let them guide the work. With equal but opposite emphasis, Russell maintained that an orderly program of materials development would be preferred to one that was directed toward "a specific and difficult application."

Stepping slightly aside from the defined field of interest, Rice opined that some nonmechanical applications, e.g., acoustic, optical, or electric functions, may be of equal or greater promise than are structural uses.

Chapman, who should know, pointed to the paucity of research on the unidirectional solidification of all-ceramic systems. He suggested that some oxide/oxide composites may have potential for high-temperature structural service. With closely similar

reasoning, Tressler recommended the study of directionally solidified ceramic eutectics and ceramic/metal eutectics, and added a suggestion for analogous exploration of very high-temperature eutectoids.

3. Prospects

Opinions expressed about the outlook for ceramic-matrix composites were hardly rosy. Writing of a type of composite that has received considerable attention, Burke took a dim view of any continuous-phase ceramic containing dispersed wires or other shaped metallic phase. Tressler was no more hopeful for the concept of strengthening ceramics with high-strength fibers of glass or other ceramic to make a body for high-temperature service; he warned of degradation of the fibers during either fabrication or service. Tallan acknowledged little enthusiasm for the Si_3N_4/metallic fiber systems that his group has evaluated. He judged that CMCs would be at their best in service wherein they bore no load, and had to tolerate only limited thermal shock.

Brown said, bluntly, that many CMCs "just are not practical." Like Tressler, he knew the danger of unfavorable alteration of properties during processing. Krochmal, who considered the development of a useful structural CMC to be a "very difficult possibility," thought the outlook bleak when he reviewed the field in 1967, and seemed of the same opinion in 1977.

Probst et al., not altogether pessimistic, thought it difficult to estimate the potential of CMC unless one had in mind a specific application, for which all conditions were defined. Warren judged that, because we are accustomed to "a ductile world," the structural use of CMCs will not come about until designers have learned to think of themselves in "a brittle world."

Davidson believed in a structural future for CMCs, and he had hope that the reliability of such systems could be brought to levels that would satisfy the design engineer.

IV. SUMMARY OF WORKSHOP DISCUSSIONS

A workshop to explore the state of technology and potential applications of ceramic-matrix composites was held at IDA on 8-9 September 1977. The attendance was made up of about 15 scientists and engineers who represented government, universities, industry, and research institutes. However, the participants were not picked by affiliation but rather by their recent interest and activity in the specific field of ceramic composites. In this way, little general background coverage was required and the workshop could get right down to specifics.

The full workshop lasted for 1 1/2 days. The subject matter covered is outlined in Appendix A, which includes copies of pertinent vugraphs and slides used in presentations. No formal papers were requested of the speakers since the purpose was to hold discussions, not a colloquim.

Immediately after the end of the workshop, a small review panel met separately to critique the workshop proceedings. This panel was made of Arden L. Bement (DARPA), Winston Duckworth (Battelle), Robert A. Lad (NASA-Lewis) and the authors. This meeting was taped and a reorganized and edited version of the tape transcript is given in the following material.

A. PHILOSOPHIC CONSENSUS

With apparent unanimity, the panel agreed that, in the promotion of the infant field of ceramic-matrix composites (CMC), an early step must be to identify appropriate applications. Once a useful application is recognized, need will then guide the endeavor to the necessary developmental research, as well

as to any basic research that still is required. In this vein, the concept of the inverted pyramid was offered: one might begin at the apex with basic research, and work toward application and eventual production at the broad base; but, during that progression, recognition of valuable potential applications would reveal that specific researches had yet to be done before development could successfully be completed. A corollary of that reasoning is that one must beware of plunging into development before the essential fundamental knowledge is at hand, or before adequate research has been done.

On the other hand, it is difficult to excite users or designers to give any encouragement to the support of basic research before the promising performance of a primitive product has been realized. If one can demonstrate the validity of a concept, and thus can tie a research program to a specific end use, then funding for the necessary basic research will more probably be obtained.

B. DESIGN, DATA BASE, AND PROGRESS OF TECHNOLOGY

Heretofore, more R&D effort has been exerted on CMCs at the processing/property interface than at the design/property interface. The designer must become more actively involved. He, working with the materials engineer, must determine the rules of design, and whether the available data base is significant for a selected application. Without valid date and without such cooperation, it is fruitless to ask the designer to design for brittle CMCs.

The decision to use a CMC for a given purpose might be based upon one of the following reasons: (1) the job could be done in no other way; (2) the consequent technology, or perhaps simplicity of design, would eliminate some muddling procedures; (3) the cost of manufacture would be reduced through the use of the special material or an advanced design. Before offering

a CMC as a replacement for another material in an existing technology, one should learn the weaknesses, if any, of the material currently in use.

Substitution by CMCs will be highly rewarding whenever it eliminates or substantially diminishes a requirement for cooling, or permits operation at a higher temperature than had been possible. A successful CMC substitute for Si_3N_4 or SiC in the ceramic gas turbine would provide a favorable cost/benefit ratio, although the material might have to be coated or otherwise protected against the combustion gases. A composite possessing significant damping capacity might well be superior to the monolithic ceramic blades of the Garrett engine, which seem to be subject to failure by vibration.

C. APPLICATIONS

On the problem of identifying applications, the panel seemed to agree that, besides discerning that a particular CMC would appropriately fulfill a recognized need, one might also perceive potential uses that would exploit the estimable properties of an available CMC. Possible applications, moreover, could be grouped into three technical categories: (1) improvements on existing ceramic components; (2) existing technology that requires better materials (but here, again, one must be certain of what is now unsatisfactory); and (3) new engineering concepts.

The specific applications that were mentioned, and were sometimes discussed at length, ranged from uses that have already been brought into practice to others that barely surpass the status of speculative suggestion. Thus, it was firmly noted that both the heatshield and the external thermal insulation of the still-developing space shuttle are ceramic-matrix composites, whereas the use of CMCs for enhanced fire protection in aircraft, in general, was merely a suggested possibility.

Reinforced concrete was strongly recommended as of great potential value to the DoD, for example, in the hasty construction of aircraft runways over an inadequately prepared subgrade; in the repair of bomb craters; in the building of certain types of boats and barges.

The promise of dimensional stability led to the suggestion of CMCs for the substrate of large mirrors, particularly those comprised by optical systems serving in space. Likewise, radomes, which require strength, erosion resistance, dimensional stability, as well as transparency to electromagnetic radiation, were a suggested application.

Several applications that were discussed had to do with the storage or the conversion of energy. It seemed obvious that a flywheel of lightweight CMC, if of sufficient tensile strength, would excel any metallic wheel in the storage of energy, because of the higher rotational velocity that would be possible. In the now-developing gas turbine built of ceramic components, turbine blades of the stator, made of Si-SiC, have done well in tests, but the rotor is still in question: no ceramic or CMC of adequate tensile strength at the target temperature has yet been developed. In such engines' combustors, however, SiC is already in use, and a CMC of appropriate design might be even more successful. It was suggested that a glass-matrix composite might well be satisfactory in the compressor fan of the gas turbine, although certainly not in the hotter zones of the engine.

Reciprocating engines offer a number of possible applications for CMCs. In the diesel, monolithic ceramics are presently under test as cylinder sleeves, piston caps, and components of the cylinder head. A major potential advantage of the nonmetallic members is that they will require little, if any, cooling. The question remains, of course, whether CMCs could provide any cost/benefit advantage over the monolithic materials. The

Stirling engine, long known but not yet significantly used, was named as an especially promising beneficiary of structural ceramics and, therefore, possibly of CMCs. The potential for the use of CMCs as bearings in propulsion systems was emphatically cited. Indeed, a fairly wide use of CMCs for the saving of weight by the automotive industry was foreseen.

A critical function mentioned as a possible application for CMCs was that of nozzles for chemical lasers, especially those to be used in space. Their use would obviate the weighty equipment necessary for cooling the currently used nickel; they could also operate at a higher temperature, and thus with greater efficiency than is now feasible. They must tolerate fluorine, to which no material seems inert at the operating temperature (about 2000°F); but, inasmuch as the total period of service is probably less than an hour, some reaction is acceptable.

Experiments on ceramic liners for gun tubes are under way, and somewhat more work has been done on rocket motors. Here, again, CMCs may be as good as or better than the monoliths. Much R&D remains to be done. Likewise, the future role of CMCs in lightweight armor remains in doubt.

D. SYSTEMS AND MATERIALS

Little attention was given to materials themselves. The question of availability, particularly of strategic materials, was once raised, but the reply, which stood unchallenged, maintained that availability is a function of price, that even strategic materials can be obtained if the price is high enough, so that no absolute shortage of materials exists.

The panel talked at length about significant or critical properties to be sought in CMCs, but they discussed only a minor fraction of the many specific systems that have elsewhere (the survey) been suggested for identified applications, or for

further R&D. High on the list of critical properties was dimensional stability, recognizable as minimum thermal expansivity in the temperature range of service. If not isotropic in that characteristic, the system should at least be symmetric in two dimensions. Other bases proposed for separating successful from unsuccessful systems included stoichiometry, toughness (whatever that is), and micromechanical attributes; the need for toughness was repeatedly emphasized. Further promise of success was said to lie in the possession of temperature tolerance (i.e., refractoriness) or a high strength-to-weight ratio. But it was also emphatically averred that the most useful system for a given service would often be one that offered something more than structural reliability, providing instead one or more valuable physical properties (e.g., thermal conductivity, piezoelectric character, thermionic emission) *combined* with strength, refractoriness, and the like.

The group favorably regarded Rice's concepts for increasing fracture toughness without decreasing fracture strength. One of his ideas was to ensure homogeneity of microstructure. Another was to augment toughness by providing small particulate inclusions (small with respect to representative flaw size), closely spaced, to deter crack propagation through "line tension" at the leading edge of the crack.

In a further discussion of the role of microstructure and micromechanics of CMCs, subcritical crack growth, which is ever a threat to materials serving under stress at high temperatures (e.g., the Garrett engine at 2250°F), was explained as accomplished through grain-boundary sliding, which generated "essentially Zener cracks" at triple points.

In yet another reference to the importance of microstructure, it was maintained that CMCs probably have significant potential for service at unlubricated points in machines rotating at very high speeds (e.g., the Garrett engine again); that, whereas the

life of the materials presently used greatly depends upon the attainment of a highly polished surface, a properly chosen CMC system, with appropriate control of microstructure, might make unnecessary most of the now indispensable polishing.

Specific categories of systems mentioned covered a wide range of temperatures. We have CMCs for use at atmospheric temperatures (e.g., reinforced concrete), for elevated temperatures (glass-matrix composites), for high temperatures (silicon nitride), and for very high temperatures (carbon/carbon, and carbon/metallic carbide). Reinforced glass was judged probably advantageous over composites with an epoxy matrix for intermediate temperatures, because the epoxy is sensitive to water; it is softened by a laser beam; and its expansivity is a major problem. Moreover, the glass-matrix composite can be hot-formed.

Reinforced concrete, of course, is already used in enormous tonnage, yet it might well be substantially improved through research. For high-temperature service, refractory concrete reinforced by stainless steel wires is in use, the wires serving principally as crack arresters.

The system Si-SiC, if the Si is held to the range of 5-10 percent, is promising for service at rather high temperatures; but the Si will creep if the system is used under stress at a temperature approaching the melting point of Si, 2570°F. [By way of a marginally appropriate comparison, it was reported that hot-pressed silicon nitride shows no creep at 2250°F.]

The external thermal insulating panels on the space shuttle were said to be made of silica fibers in an oxide. The composition was not more precisely given, and was thought probably proprietary.

Aluminum nitride was mentioned as very interesting, but no one proposed a composite system of which AlN would be a component.

E. FABRICATION

CMCs are often made by use of classic ceramic techniques for the compaction of powders with reinforcing particles or fibers: (1) cold-pressing, followed by sintering; (2) hot-pressing; or (3) hot isostatic pressing. A variation on pressing is explosive forming in which the employment of a graded powder was said to help to absorb the energy of the shock wave and to prolong the period of high pressure ("high-pressure dwell time").

It was emphasized that the assembly of dissimilar materials in the above manner is usually best done at the lowest feasible temperature so that damage of the components by high-temperature reaction may be avoided. In any event, the movement or transfer of materials, whether in the initial combination or in a subsequent processing, and the related control of dimensions, are always critical considerations.

For a limited number of systems, a sol-gel source for at least one material may be utilized. When the gel is dried, it may be reduced to powder (or spherical granules). The necessary chemistry is often intricate but that impediment is largely compensated by the simplified chemical engineering and handling. Any impurities present are not segregated but are almost atomically dispersed. There are no concentration gradients, and no contaminating comminution is required. Final processing is accomplished by conventional techniques.

The preparation of microcomposites by directional solidification of eutectic melts, a scientifically fascinating technique, was judged to be of limited applicability. If the fibrous component is a refractory metal, the system will have poor resistance to oxidation. With a suitable nonmetallic fiber, the threat of oxidation could be avoided, but the consensus was that, for any system of this type, scale-up to practice would be very difficult.

The several techniques for fabrication, machining, etc., of CMCs are usually available, but economic feasibility often determines the choice (and the exclusion) of techniques; whether preparation shall be done, for example, by sintering, by chemical vapor deposition, or by explosive forming; and how much machining shall be done, and whether, e.g., ultrasonically or by diamond grinding.

The workshop brought out a better understanding of fracture surface energy, and of how that entity is affected by microstructure and by a second phase--hence, by fabrication.

In the discussion of fabrication, it was more fully brought out (see Systems and Materials in Section D above) that control, not only of constitution, but of microstructure as well, may permit the fabrication of bearing surfaces without all the grinding and surface preparation heretofore required.

Strengthening of certain systems may be done by application of compressive force, e.g., in machining, to induce transformation in the surficial layer of one component. It was suggested that ion-implantation, for which equipment will soon be commercially available, could do much the same thing.

F. SOME COMMENTS ON R&D

A few of the topics discussed by the panel on general R&D problems deserve attention.

1. *Defining research problems* for investigation requires the establishment of aims. A substantial part of fulfilling that requirement consists of learning the customers' needs; that is, see what the applications people want.
2. The panel clearly agreed that the aim of basic research is to generate understanding. To formulate a good fundamental research program is to identify some principal areas that are judged to be potentially useful. Several such specific subjects follow.

a. How much effort should be devoted to the derivation of phase diagrams? (The need for such information frequently surfaced during both the workshop and the critical review thereof.) Such a program was adjudged most likely to be approved for funding if specific practical needs could be demonstrated. The best approach was thought to be to identify small critical points of constitution for which knowledge is lacking, and to seek support for these narrow areas which impede progress.

b. The mechanisms whereby fracture surface energy may be increased are poorly understood. While that lack of understanding endures, progress in the field will be confined to the fruits of trial and error.

c. Test methods, based upon fundamental principles, are needed for CMCs exhibiting toughness. How does one analyze such a fracture?

d. Presently available methods of mathematical analysis of the propagation and reflection of an elastic wave in armor are said to be quite inadequate for the treatment of data and the prediction of behavior of projectiles in an ongoing penetrator program. The same weaknesses should apply to any macromechanical study of CMCs.

3. Two subjects were suggested as appropriate for work directed toward specific application.

a. Uses should be sought for fiber-reinforced glass.

b. The field of radomes and IR domes seems a promising one for ceramic and CMC research.

V. FINAL ASSESSMENTS AND RECOMMENDATIONS

A. ASSESSMENT OF FINDINGS

Previous sections of this paper have attempted to summarize the views expressed in the literature (or in private discussions) concerning ceramic-matrix composites, the views expressed by the respondents to a broad survey of ceramic scientists and engineers, and the deliberations of the CMC Workshop. We shall now attempt to condense this information and assess it in the context of the task objectives. To a certain extent it is recognized that some of the statements made herein are assertions or opinions, especially since there have been few, if any, detailed applications tradeoff studies. However, it should be quickly reemphasized that these are not the assertions of the authors, alone, but include critical consideration of the opinions of the technical community. In the case of structural ceramic-matrix composites, it is not a matter of assessing the efficacy of what the DoD is presently doing; essentially, there is no ongoing DoD program of sufficient size to warrant a critique. Rather, then, what is being done is to assess the future potential of CMCs, starting from practically a zero present applications base, which must necessarily involve educated opinions as the initial step.

1. Potential Usefulness of CMCs to DoD Systems

The potential DoD applications interest in CMC material systems is very broad, including every area where structural ceramics are of interest and some where monolithic ceramics would not even be considered. While prior discussions in this

paper have divided the application potential according to temperature scale, here the division will be on a short-term (5 years or less) and a long-term usefulness. This judgment, of course, is intertwined with the following section's assessment of technology status.

a. <u>Short-Term (<5 years) Applications</u>

<u>Seeker Domes.</u> Many of the present radomes are made of slip cast fused silica which presents problems in both integrity and rain erosion. There has been past work on quartz filament-wound silica materials which showed promise. Whether silica is sufficient is open to question, but a strong composite effort would seem reasonable.

<u>RV Antenna Windows.</u> Present quartz/silica or boron nitride/boron nitride are marginal performers, as pointed out in an earlier IDA paper (Ref. 1). DoD work on high-performance boron nitride fibers is almost at a standstill (as well as the composite) and should be reinvestigated for this as well as other applications.

<u>Concrete Structures.</u> Iron wire-reinforced concrete is almost reaching a commercial stage, particularly through the efforts of Battelle on selling their Wirerand® process. The DoD (especially the Army Corps of Engineers) has been somewhat remiss in not applying a larger effort to investigate the potential uses of this and similar reinforced concrete (or cement) composites. In particular, the Navy (in spite of a flurry of effort to design conventional handwoven and tied ferrocement boats to be built by low-cost-labor countries like South Vietnam and South Korea) has not appeared to have given any serious attention to castable ocean vehicle hulls using such composites.

<u>Armor for Kinetic-Energy Penetrators.</u> The analytical ability to predict how successful armor arrays should be built

is presently very limited. A systematic experimental program to empirically outline the materials systems needed appears to be in order. One supposes, with some confidence, that reinforced ceramics (including laminates in this case) will have a high priority. However, here is a situation where one still does not know what the parameters should be, except in very general terms. The military systems problem is quite pressing.

Moderately Increased Temperature Gas Turbines. A moderate increase of uncooled gas-turbine inlet termperatures to 2000°F (with excursions up to about 2200°F for short periods) may be feasible using silicon carbide fiber/silicon composites. While obviously not as exciting as temperatures of 2500°F or more, such applications do offer the promise of increased fuel savings and perhaps at low cost (by comparison with superalloy materials). Present DoD ceramic turbine efforts are concentrating almost entirely on the higher temperature materials. While this should be done, it would seem that effort on a near-term practical application would be worthwhile. It would also give ceramic use a chance to walk before it runs.

Erosion-Resistant RV Nosetip. This is a seemingly pressing problem, which has been covered in a previous IDA paper (Ref. 1). The emphasis here is that the tantalum carbide/graphite composite (Appendix A, page A-15) and the boron carbide/graphite composite (Appendix A, pages A-40 and A-42) are promising enough that they should be considered strongly for applied system efforts.

Shipboard Multifunctional Incinerator. An ongoing Navy 6.3 program, this device is having serious refractory liner problems due to the various criteria it must meet, not the least of which is being usable on board a ship (Chapter II, Section D-4). The Navy is presently attempting to use a thin castable refractory lining either sprayed or (more usually) troweled in place. It would appear feasible and advantageous to incorporate

ceramic fibers (perhaps alumina or silicon carbide, depending on the precise matrix). Here is a case where the initial material cost, while not negligible, is not a paramount factor if greater relaibility can be achieved. See also Ref. 8.

b. Long-Term (>5 year) Applications

Reinforced Glass Composites. Although not yet designed for a specific application, this type of material now appears to have sufficient basic data to indicate a high promise and to warrant a search for usefulness. The reader's attention is directed to Appendix A, Section 6. With a density close to that of carbon-reinforced/polyimide (and much less than that of carbon-reinforced/aluminum), carbon-reinforced/glass has a potential use temperature of over 1200°F as opposed to 700°F-1000°F for carbon-reinforced/aluminum. Recent developments seem to have alleviated or removed (perhaps with the use of recently developed silicon carbide filaments) the problems about oxidation resistance of the fibers (see Ref. 9). The glass composites have strengths, toughness and stiffness at least equal to the organic-matrix composites, are potentially as cost effective, and have better water vapor resistance. They (glass composites) have the potential of being readily hot-formed to complex structural shapes. This is a new class of advanced composites which complement the organic and metal matrix composites. Although the present study has not looked hard at detailed applications, such investigations should be made. Note that elevated temperature use is not necessarily the most beneficial application. Good dimensional stability (including low thermal expansivity), for example, suggests use in large space mirror substrates.

Air-Breathing Engines. From a resources standpoint, the bulk of present DoD ceramics R&D lies in support of the ceramic gas turbine, and almost all of this effort is concentrating on silicon nitride and silicon carbide. The allowable

uncooled turbine inlet temperature of 2500°F is the temperature value usually quoted by propulsion designers as the dividing line of interest. There is little competition from concepts involving metal turbine redesigns at this temperature and higher; ceramics have to be used and the benefits from reduced specific fuel consumption (among other factors) are great. Below this temperature (down to about 1900°F), ceramics would be useful but cost enters in more strongly; in this regime, metals can be used, not as well but perhaps well enough. A demonstrator test with a silicon nitride rotor has been successfully run for one hour, at about 50,000 rpm, at 2500°F, which shows that it can be done. However, as emphasized throughout this paper, this is not really the problem. The question is whether [through proper nondestructive evaluation (NDE) or an improved ceramic] the material components can do this every time and, of course, for at least a duty cycle of 200 hours. In addition, foreign object impact damage still needs much more attention (although clearly of greater importance to a jet engine). Most members of the ceramic community have expressed concern as to whether a monolithic ceramic can be successful (at least in taking full advantage of ceramic properties) for this application. This area engendered the largest number of suggestions where it was thought that CMCs ought to be researched.

Diesel engine designs are well along the way to using ceramics for their refractoriness and thermal insulation, with an eye toward reducing or eliminating cooling requirements. This goal is well worthwhile in itself. However, further significant advances (particularly in areas of reduction of energy use) can evidently be made by making all-ceramic hot moving parts, thus taking advantage of the higher ceramic strength-to-weight ratios. To do this would probably require composites, but the design problems do not seem to have been explored (unless by the private automotive firms).

Preliminary designs for a closed-Brayton-cycle ship propulsion engine (Chapter II, Section D-2) indicate that if a ceramic heat exchanger could be built to operate for long periods at 2500°F, the specific weight of the power plant could be reduced by as much as a factor of ten. Such a heat exchanger would see fossil fuel combustion products on one side (an inert gas on the other) and must run at this temperature for about 10,000 hours (although not continuously) without need for serious repair. While a stationary device, the long-term thermal stresses and oxidation present extremely difficult problems. In similar reasoning as for DOE's (Department of Energy) stationary coal-fired ceramic power-plant concept, many members of the ceramics community have expressed concern about long-term oxidation in silicon nitride and silicon carbide. They would feel much easier, from this standpoint, if components were built of oxide materials for such long-term use. However, oxides would almost surely have to be toughened by compositing. This is a strong argument for concentrating some R&D effort on oxide-matrix composites, especially since it could also apply to short-use-cycle-time engines.

c. Other Applications. There are a number of other less well-definable CMC applications (see Appendix A, Section 9). Two that should be mentioned here are ceramic bearings (most likely silicon nitride) and ceramic gun tube inserts (probably alumina or zirconia). Another longer term use might be nozzles for chemical laser systems.

2. The Present Technology for CMCs

On balance, our understanding of the technology of most ceramic-matrix composite systems is increasing although still incomplete. Generally speaking, innovative ideas are still required, even though enough about the basic mechanisms has been learned over the last few years so that the kinds of ideas needed

can be fairly well defined. In other words, we now pretty well know where we would like to go, but are not sure of the best way of getting there. The situation is not unlike the very early days of high-performance organic matrix composites; the potential advantages were known but the understanding of problems in chemistry, physics, and the mechanics of fabrication was weak.

Nonetheless, there have recently emerged a few ceramic composites which are far enough advanced, or look so good, that they are candidates for a 6.2 (or even 6.3A) effort. In every case, as pointed out above, a deeper application benefit analysis should be undertaken. The most obvious such composite is fiber-reinforced concrete or castable refractory. Battelle has developed an iron wire (chopped) concrete to the point where large-scale demonstration efforts on roads and bridges are under way. This, or perhaps a continuous woven wire variation, could be used, as an example, for experimental boat hulls. Ceramic fibers (glass or alumina) have not been worked on as much, but there is every reason to think they could be used for near-term application where oxidation precludes the use of iron (there may, however, still be some problem about chemical interactions between glass and Portland cement on curing). Refractories for ship incinerators deserve special attention.

For aerospace uses, tantalum carbide fiber-reinforced graphite is in good technological shape. Although not as far along, particulate composites of boron carbide and graphite look promising. An immediate application for either would be erosion-resistant ballistic reentry vehicle nosetips. Although a complicated system, various forms of silicon/silicon carbide/carbon composites have reached a stage where consistent and economical processing can be accomplished. They may have near-term usefulness in moderate increases of gas-turbine inlet temperatures. There is a promising possibility that silicon nitride can be

surface toughened by a cold work process (in effect, this appears to be particle compositing by stress-induced phase precipitation). This could be very useful for cold applications such as increasing the reliability of ceramic roller or ball bearings. Finally, continuous silica or alumina fibers in a silica or alumina matrix have had enough previous R&D that the concept out to be exhumed for radome use.

The last CMC system to emerge from this study, and which is probably a 6.2 candidate, is a glass matrix reinforced with continuous fibers of boron, graphite, or silicon carbide. The remarkable set of properties which can be obtained from this CMC is discussed earlier in Section 1 of this Chapter (see page 54) and will not be repeated here. The Review Committee of the IDA Workshop was unanimous in saying that this class of CMCs be given a very high priority for near-term DoD development.

Most of the existing problems for CMCs fall in the 6.1 category. However, there are some negative conclusions reached by the workshop which are useful.

- Metal (especially refractory metal) fiber-reinforced ceramics are not promising for any high-temperature application in an oxidizing atmosphere. It does not matter whether the metal is grown *in situ* or not.
- Purely exploratory work searching for new chemical composite systems no longer seems justifiable. It is time to narrow down efforts to a few chemical systems and understand their fabrication potential and properties.
- Typical hot-pressing of refractory ceramic powders with blended-in brittle fibers is likely an unpromising fabrication technique. A (positive) corollary of this, particularly for oxides, is that directionally solidified ceramic fiber growth techniques should be emphasized.

Despite an impressive accumulation of knowledge, the mechanisms of toughening and strengthening (which, in general, are two different things and frequently act in opposite directions) are still not sufficiently clear in ceramic-matrix composites. The most commercially successful CMC, partially stabilized zirconia, is not really understood except in rather vague terms (Ref. 10). Toughening is apparently due to stress-induced metastable phase transitions, and seems to work only when the high-temperature phase has a lower density than the stabilized low-temperature phase. Whether the effect is limited to zirconia systems is not clear, but the general phenomenon of stress-induced phase transitions deserves a specific study.

Directionally solidified (DS) growth of oxide fibers or lamellae in an oxide matrix has been successfully demonstrated in several materials systems (see Appendix A, Section 2). This is presently a long slow process and it has often been suggested that scale-up problems may preclude such CMCs from being practical. While this is possible, the present study seems to indicate that such *in situ* growth may well be the *only* way in which useful high-temperature oxides can be ceramic fiber-reinforced. Certainly it has the potential for yielding excellent microstructure configuration. While previous DS oxide/oxide R&D has been good work, it has not been intensive and very little effort has been devoted to fabrication techniques as such. This seems important enough to warrant a broader study.

Silicon nitride and silicon carbide have had the heaviest attention, over the past 5-7 years, of any of the monolithic ceramics. This is perhaps as it should be, but there have been numerous expressions of concern that such monolithic materials might not be able to fully satisfy the needs of their strongest driving application, namely the gas turbine engine. Compositing them with ceramic fibers would seem to have a very high priority, but no one is quite sure how to do it. In 1970, Lindley and

Godfrey (Ref. 11) reported results of putting long (but discontinuous) silicon carbide fibers into a silicon nitride matrix. The results looked good; the fracture surface energy was in the range of cast iron. However, nothing more appears to have been done perhaps because their silicon carbide fibers were vapor deposited on a tungsten substrate. As pointed out elsewhere, such a fiber encounters thermal stability problems around 850°C and cannot be used to higher temperatures. It is possible work is under way in the United Kingdom which escaped the attention of the authors. In any case, better silicon carbide fibers are now available and a new look ought to be taken. In 1968, the AFML carried out a short research effort (at Stanford Research Institute, Ref. 12) to grow *in situ* silicon nitride whiskers in a silicon nitride matrix. The work was dropped because high density bodies could not be obtained. However, the concept may well be feasible and certainly another study should be made (the AFML project had little publicity and none of the workshop participants was even aware of it).

A seemingly promising area of research for silicon nitride (and maybe silicon carbide) is to look for appropriate ways of making sols and gels perhaps similar to the way silica gel is made. The advantages can be very high (see paragraph above) provided it can be done. It is not immediately obvious that the necessary chemical precursor compounds exist that would permit silicon nitride to be precipitated from a room temperature solution. Perhaps an arc discharge technique in liquid ammonia using silicon electrode might work (similarly to how colloidal gold precipitates are made), but it apparently has never been tried. There was really no one involved in the workshop who was experienced in silicon-nitrogen chemistry and the authors feel that an appropriate consultant panel ought to consider the question more deeply.

It appears to the authors (and others) that the corrosion properties of silicon nitride and silicon carbide have not been

sufficiently researched. These materials depend for their oxidation resistance (other than merely decreasing porosity as much as possible) on a self-healing film of vitreous silica (or, more generally, silicates). This, of course, is also the oxidation resistance mechanism of silicides. There are known difficulties, at certain temperature regimes (e.g., quite low temperatures), with the oxidation of both silicon carbide and molybdenum disilicide. Manufacturers of silicon carbide furnace heating tubes carry a warning to the user not to run them very long (in air) at temperatures around 900-1000°C (1832°F). Above and below this temperature range the oxidation resistance is good, but in that range the silicon carbide will oxidize reasonably rapidly. Such a phenomenon also exists for silicon nitride though no publications were found. This whole subject may or may not be a problem in gas turbine applications but it does not appear to have been very deeply considered (considering its potential implications).

A very significant area for 6.1 research attention is that of innovative CMC processing techniques. *In situ* DS ceramic fibers in a ceramic matrix have already been mentioned in the context of oxide/oxide composites. As a broader statement, attempting to hot-press fibers (nonmetallic) in a refractory ceramic powder will generally not work well enough to ever warrant consideration as a manufacturing method. Various possible methods are discussed in Appendix A, Section 8. Some gas-solid (or liquid-solid) reaction techniques are already being used for the monolithic ceramics. Others, such as *in situ* pyrolysis (or copyrolysis) of precursor polymer fibers, are really just concepts. Sol-gel powder preparation to form very fine (almost atomic) dispersions of one material in another looks promising. High-pressure, high-strain-rate (e.g., explosive) forming has never been seriously applied to ceramics, partially because of cost. However, the workshop panel agreed that such techniques have high potential and that joint programs

between DoD and DOE (which is where the expertise presently exists) should be seriously contemplated.

B. RECOMMENDATIONS FOR DoD ACTIONS

From the discussion of Section A, there is left little doubt that the potential usefulness of CMCs to DoD systems is high and very broad in scope. With a few exceptions, it is not quite so clear which areas of technology are likely to prove the most promising for instigating R&D programs. In Option I, below, the authors have attempted to lay out one suggested program involving what appears to be the smallest and most effective investment of resources. Option II lists additional desirable projects if (or when) greater resources become available. Since CMCs, as a whole, form a cohesive technology with common basic problems regardless of the specific materials involved, it is strongly recommended that the program be organized as a single coordinated DoD effort to permit the best use of funding.

OPTION I: SUGGESTED MINIMUM CMC PROGRAM

No attempt will be made to lay out a schedule or to delineate between in-house or contract work. Since almost all projects would be new starts (even if serving the needs of an ongoing program), only the first year's efforts is indicated. It is understood that each project would be reviewed periodically with the direction or funding altered as appropriate. The suggested projects are listed in order of closeness to applicability and seeming potential. Estimated resources required for the first year, in man-years (MY), are shown for each. These estimates reflect a minimum meaningful project size based on the authors' background experience.

1. Near-Term (<2 years)

1.1 Ceramic Fiber/Castable Refractory. To investigate chopped alumina fiber in typical high alumina refractory liners for use in newly developing Navy shipboard multifunctional incinerators. (3 MY)

2. Moderate-Term (2-5 years)

2.1 Nonmetallic Continuous Fiber/Glass. To investigate boron, carbon, and silicon carbide fibers imbedded in various glass matrices. An intensive applications tradeoff study is suggested as part of this. An existing Air Force project is about a one man-year per-year-effort on alumina fibers for possible radome use. (2 MY)

2.2 Erosion-Resistant RV Nosetip. To investigate further existing carbide/graphite composites developed by Los Alamos and by NRL for reentry vehicle nosetip application. There is a small existing Navy contract, estimated at about one MY per year. (2 MY)

2.3 Oxidation Resistance of SiC and Si_3N_4. To investigate the stated problem so that reliable projections can be made to 10,000 hours. Also, the question of short-time low-temperature (about 900-1000°C) should be resolved. While not strictly a CMC project, the results could have a strong impact on the urgency of future CMC investigations. (1 MY)

3. Long-Term (>5 years)

3.1 SiC and Si_3N_4 Fibers/Si_3N_4. To investigate the use of the newer SiC long fibers in a Si_3N_4 matrix. In a concurrent part of the project, investigate the *in situ* incorporation of fibrous or whisker-like Si_3N_4 in the same matrix. (2 MY)

3.2 Oxide Fibers/Oxide. To investigate further *in situ* DS oxide growth in oxide matrices with special attention to

improved processing techniques. This is an important backup technology in case SiC and Si_3N_4 prove impractical. (2 MY)

3.3 <u>Sol-Gel techniques for Carbides and Nitrides.</u> To explore innovative sol-gel methods for precipitating (or coprecipitating) very fine particle composites, particularly relating to SiC and Si_3N_4. The Navy has some sol-gel in-house research. (1 MY)

3.4 <u>*In situ* Copyrolysis of Fibrous Precursors.</u> To explore innovative methods whereby reinforcing fibers can be formed in place (within the matrix) by pyrolytic techniques. The Navy has a small in-house effort. (1 MY)

3.5 <u>Toughening and Strengthening Concepts and NDE.</u> To better understand the micromechanics of CMCs and potential NDE techniques. The Navy has an in-house effort. (2 MY)

The total estimated resource effort required for Option I is 16 man-years for the first year.

OPTION II: SUGGESTED DESIRABLE CMC PROGRAMS

This option includes Option I and the following suggested projects.

1. <u>Near-Term (<2 years)</u>

1.1 <u>Metal Fiber/Concrete.</u> To investigate and expand on existing iron or steel-fiber-reinforced concrete for possible application to castable boat hulls. (2 MY the first year)

2. <u>Moderate-Term (2-5 years)</u>

2.1 <u>Silicon Carbide/Silicon/Carbon.</u> To investigate the application of existing technology of either preform SiC/Si or molded SiC/Si/C materials for medium-temperature gas turbine engines. (2 MY)

2.2 <u>CMCs for Gun Barrel Liners.</u> To investigate the various concepts, and their potential benefits whereby CMS could

be applied to the gun barrel erosion and wear problems. (1 MY the first year)

2.3 CMCs for Ceramic Bearings. To investigate concepts whereby ceramic roller and ball bearings (especially Si_3N_4) might have their life and reliability significantly increased by the use of CMCs. (1 MY the first year)

2.4 Boron Nitride Fibers/Boron Nitride. To investigate techniques of producing high-performance BN fibers and incorporating them into a dense BN matrix. The first application would be RV antenna windows, but there will be others depending on the results. (2 MY)

2.5 Alumina, Silica Fibers/Alumina, Silica. To reinvestigate the fabrication of filament-wound reinforcement in a silica or alumina matrix for erosion-resistant radome use. The fibers could be quartz or alumina. This could be a 6.3A project. (2 MY)

3. Long-Term (>5 years)

3.1 CMC Fabrication Techniques. To investigate the utility of innovative CMC fabrication methods. This would include a more thorough look at flame spraying, plasma spraying, chemical vapor deposition, explosive forming, etc. Expertise exists in the country for each of these methods (albeit for other uses). (3 MY)

The supplemental estimated required resources are 13 man-years, so that the total resources for Option II are 29 man-years for the first year.

If the DoD decides that the military applications described above are needed, then the recommendation is made that the DoD consider and implement one of the two suggested options.

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APPENDIX A

TECHNICAL OUTLINE OF WORKSHOP ON CERAMIC-MATRIX COMPOSITES

CONTENTS

APPENDIX A

TECHNICAL OUTLINE OF WORKSHOP ON CERAMIC-MATRIX COMPOSITES

The workshop on ceramix-matrix composites held at IDA on 8-9 September 1977 was attended by about 15 persons. This workshop was intended as an informal discussion, in depth, and not as a seminar with formal papers. Some of the attendees, however, were requested to give short talks as an introduction to specific technical areas; this appendix gives short excerpts from a few of these talks. The format varies but generally will be a short resumé (or explanatory comments) plus pertinent data (usually unreferenced) presented by the speakers. The authors made no attempt to analyze the sections; in fact, the sections are frequently left in the form of a vugraph briefing. The purpose of Appendix A is to present some of the raw information made available to the workshop participants. A short but useful outline of notes on the entire workshop is given in Section 11.

Appendix B is a list of respondents whose replies to the survey questionnaire were used by the CMC Workshop. Appendix C contains the survey questionnaire sent to over 300 persons knowledgeable in the science and engineering of high-performance ceramics.

1. OXIDE MATRICES/METALLIC REINFORCEMENT*

The problem areas associated with the use of metallic reinforcement of oxide ceramics are summarized in the following tabulations. Although the utilization of *in situ* grown metal oxide-metal eutectic structures offers some improvement over incorporating metal wires in a hot-pressed oxide matrix, the inherent lack of oxidation resistance of both materials severely limits utilization for any practical applications. These materials exhibit major improvements in impact and work of fracture values compared to pure oxides at room temperature, but no success has been made at retaining these properties at elevated temperatures (>1000°C).

*Contributor: A.T. Chapman.

Metallic Reinforcement of Oxide–Matrix Ceramics
(i.e. wires incorporated in hot pressed oxides)

GOAL: Improve Thermal and Mechanical Shock Behavior

PROBLEM AREAS:

1. Fabrication methods weaken (embrittles) metallic fibers
2. Obtaining uniform fiber distribution
3. Thermal expansion mismatch
4. Oxide-metal surface (debonding) problems
5. Flaws exceeding critical size
6. Often tensile and compressive strength of composite less than pure oxide (mullite notable exception)
7. Very little oxidation resistance

Unidirectionally Solidified Metal Oxide-Metal Composites for Structural Applications

Goal: Improved Mechanical Strength and Impact Properties

ADVANTAGES:

1. Metallic fiber distribution excellent
2. "Coherent" fiber–matrix interface
3. Single crystal "ductile" fibers
4. Less than 1 micron diameter fibers

DISADVANTAGES:

1. Fabrication requires crystal growth techniques followed by hot pressing
2. Very limited choice of oxides and metals
3. No understanding of the high temperature chemistry (metal solubility in molten oxides)
4. No oxidation resistance

CONCLUSION:

Most systems that consider ceramic components in future upgraded designs need materials that are operational at ever increasing temperatures. In no case has a metal reinforced ceramic matrix composite performed well in an oxygen environment at temperatures where the super alloys are used today. Except for a few applications (in low oxygen pressures) further R & D in this area is probably unwarranted unless some way is found to greatly improve the oxidation resistance of this class of composites.

2. OXIDE MATRICES/NONMETALLIC REINFORCEMENT*

Examination of oxide-matrix composites can be on the basis of microstructural features of the "reinforcement" phase. On this basis, the existence of a very fine or a dispersed phase is one category, a much larger particulate phase is a second one, and the fibrous or lamellar structure is a third. The dispersed versus particulate category may be divided at about the one-micron level, whereas the fibers or lamellae may be considered continuous.

The dispersed second phase in oxide composites may be considered on the basis of dislocation processes or crack processes. The use of precipitates via precipitation hardening has been unequivocally demonstrated to affect dislocation processes as increased microhardness in at least two systems--spinel and sapphire. Modest hardness increases (about 20%) have been reported in single-crystal alumina-rich spinel, heat-treated to yield a metastable Al_2O_3 precipitate. Similarly, a TiO_2-rich precipitate forms in the classical star sapphire with an attendant increase in the hardness. There is little doubt that the classical precipitation hardening process can be successfully applied to some oxide systems. It has been amply demonstrated in single crystals, but to date has not achieved any form of large-scale polycrystalline structural application.

The interaction of cracks with precipitates in spinels has also been demonstrated as they exhibited increased strength and decreased susceptibility to slow crack growth. No commercial application has been advanced.

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*Contributor: R.C. Bradt.

The interaction of cracks with a precipitate phase has also been clearly demonstrated in partially stabilized zirconia (PSZ). A zirconia body with a cubic phase stabilizer (Y_2O_3, CaO, or MgO) is heat-treated in the cubic + tetragonal phase field. Upon cooling to lower temperatures, a microstructure of cubic zirconia containing metastable tetragonal precipitates results. When a crack is stressed, the crack tip stress field causes the metastable tetragonal zirconia to transform to the stable monoclinic form. An increase in toughness results. Fracture toughnesses between 5 and 6 $MN/m^{3/2}$ have been reported, and strengths approach 100,000 psi. It is expected that this strong/tough material will exhibit enhanced properties to about 1000°C. In addition to the many research efforts concerning this concept, a related material is being marketed in the form of dies for extrusion and wire drawing. Thus it is commercially available in bulk form.

Related to this metastable precipitate phase transition in a crack tip stress field to yield enhanced toughness and strength is the development of a completely metastable body of tetragonal zirconia. Through proper additives (same as before) and small particle sizes, a dense tetragonal body can be achieved. Crack tip stress fields cause a conversion to the monoclinic form. Fracture toughnesses as high as 9 $MN/m^{3/2}$ have been reported, along with 100,000-psi strengths. Again, about a 1000°C upper limit is anticipated. No commercial forms of this have been marketed.

The incorporation of particulate oxide particles into an oxide matrix has been thoroughly studied for ZrO_2 particles hot-pressed in an Al_2O_3 matrix. Several points are clear and may be extendable to other systems. There is absolutely no doubt that crack/particle/microcrack interactions can increase energy dissipation, perhaps arresting cracks. However, particles also serve to lower strength in most cases by creating "microcracks." It is reasonable to assume that little, if any, increases in

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strength can be expected from particulate additions. Submicron particle additions may overcome these problems where particle-associated microcracks are much smaller than intrinsic micro-structural flaws.

Although not a ceramic second phase, there are two areas of ceramic matrices that merit some attention. One is polymer impregnation of concrete and/or structural clay products and the other is the use of natural structures, such as coral and wood, to develop a ceramic matrix. In these cases, the pore "filler" usually has only low-temperature capabilities and not very promising strengths, although strengths do reach two to three times their nonimpregnated values. This type of ceramic composite where both phases are continuous does have some merit for low-temperature, low-strength considerations.

Continuous fiber or lamellar oxide composites have been developed by directional solidification of oxide-oxide eutectics. These possess good hardness, wear, toughness, and creep characteristics, as well as excellent high-temperature (>1500°C) strengths. Most of the structural and many of the property characteristics have been established. For commercial use, sizes greater than about one inch in diameter have not yet been directionally solidified. Potential for the large sizes does exist at the Army Materials & Mechanics Research Center (AMMRC) facility.

In summary, promise exists for use of oxide composites in structural applications. Some outstanding properties have been obtained. Based on the metastable zirconia studies, as well as the directionally solidified eutectic studies, it appears that *in situ*-type composites offer the best promise. That is, particulate composite results suggest difficulties in taking the two components as separate entities and consolidating them. Many questions need to be answered, particularly concerning the fundamentals as well as commercial scale-ups. Some concern the

metastable phase approach. What makes a metastable phase? and Can any phase transition be used? How can some of the other composites be scaled up? Very interestingly, the only example which has "real" success and is commercial in nature, PSZ, is also the least fundamentally understood.

3. SILICON AND SiC MATRIX COMPOSITES*

SiC-Si composites' technology has advanced to the commercial state for several compositions. The United Kingdom's Atomic Energy Authority REFEL and Norton's NC 430 are available in commercial quantities with the attractive properties listed in Table I.

Other types of processing dealing with the formation of SiC fibers in a silicon matrix (Ref. 1) and with transfer molding of SiC-polymer compositions (Ref. 2) to form SiC-Si composites are in the development stage and have yielded potentially useful refractory parts for gas turbine applications. A summary of their physical and mechanical properties is shown in Table I.

TABLE I

PROPERTIES OF SiC-Si COMPOSITES (Ref. 2)

	SiC Vol %	Density g/cc	Rupture Strength (Kpsi)	Young's Modulus (10^6 psi)
Commercially Available				
REFEL Extruded	90	3.10	76	60
Norton NC 430 Slip Cast	90	3.1	45	54
In Development				
Ford Transfer Molded	90-95	3.14	70	60
General Electric	80-85	2.87	70	57
Infiltrated	40-45	2.70	48	44
Carbon	20-25	2.60	30	29

*Contributor: T.J. Whelan.

The principal advantages of these materials are that they can be formed from initially inexpensive materials to final shape with less than one percent of dimensional change during processing. Another important advantage is that they yield high strengths essentially independent of temperature to about 1300°C. Both isotropic and anisotropic strength properties can be developed by the General Electric process.

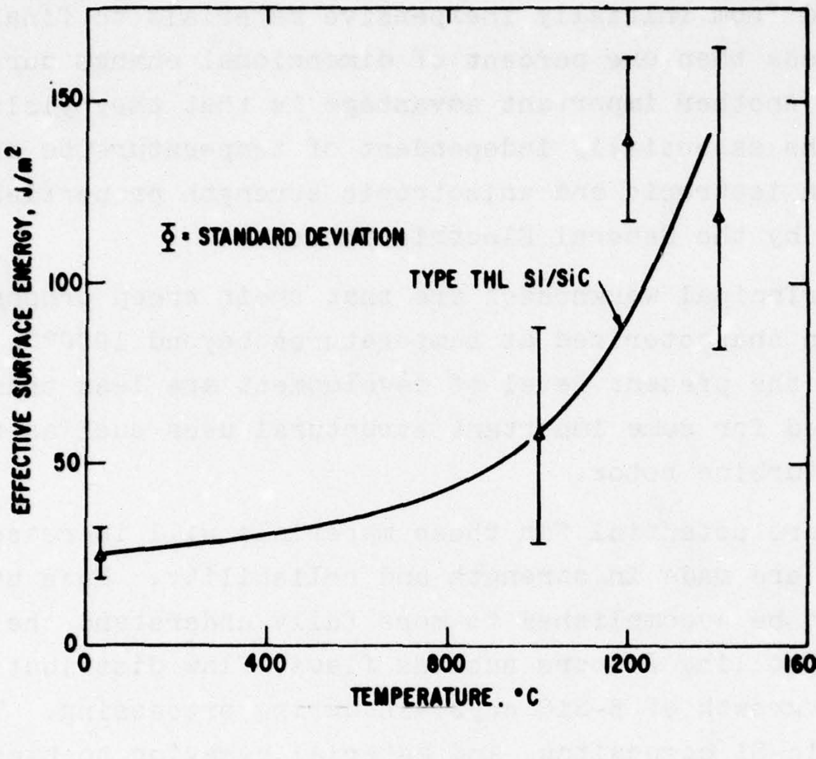
Their principal weaknesses are that their creep properties have not been characterized at temperatures beyond 1000°C, and strengths at the present level of development are less than those required for some important structural uses such as the ceramic gas turbine rotor.

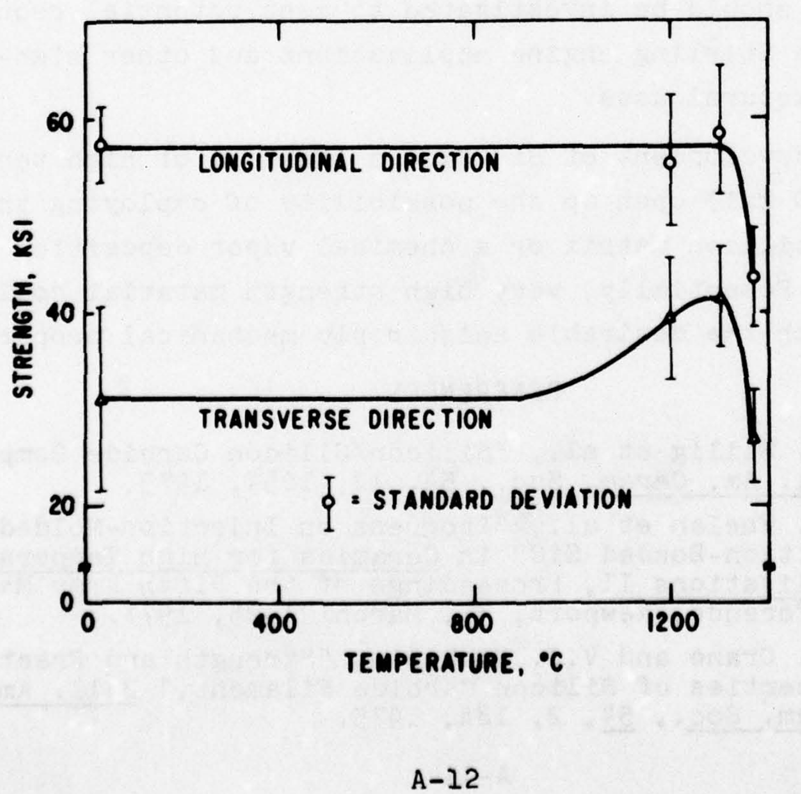
The future potential for these materials will increase as improvements are made in strength and reliability. Much basic research must be accomplished to more fully understand the strength-controlling factors such as flaws, flaw distribution, and the intergrowth of β-SiC crystals during processing. The joining of Sic-Si composites, and material behavior to high-pressure H_2, should be investigated to meet potential requirements for the Stirling engine applications and other high-temperature structural uses.

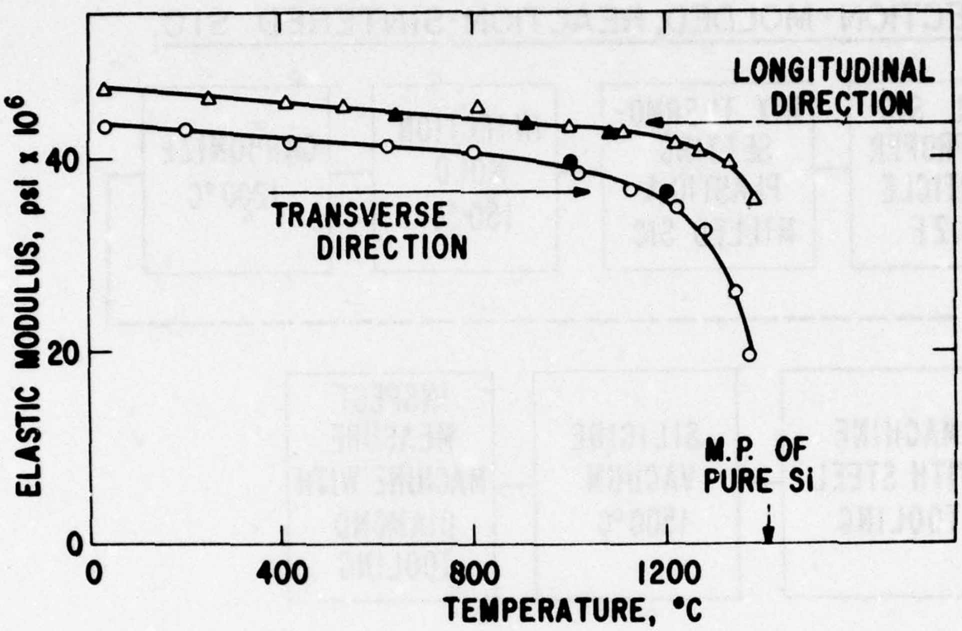
Recent development of SiC fibers (Ref. 3) of high tensile strength (300 ksi) open up the possibility of employing these fibers in a silicon matrix or a chemical vapor deposition (CVD) SiC matrix. Potentially, very high strength material could be developed with the desirable anisotropic mechanical properties.

REFERENCES

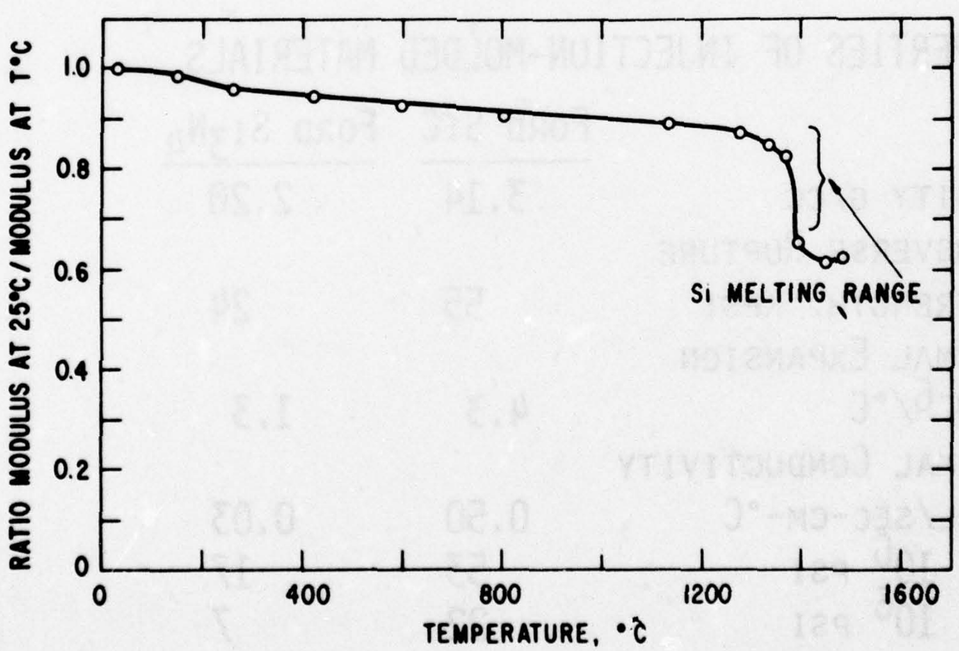
1. W.B. Hillig et al., "Silicon/Silicon Carbide Composites," Bull. Am. Ceram. Soc., 54, 12, 1054, 1975.
2. T.J. Whelan et al., "Progress on Injection-Molded, Reaction-Bonded SiC" in Ceramics for High Temperature Applications II, Proceedings of the Fifth Army Materials Conference, Newport, RI, March 21-25, 1977.
3. R.L. Crane and V.J. Krukonis, "Strength and Fracture Properties of Silicon Carbide Filament," Bull. Am. Ceram. Soc., 54, 2, 184, 1975.


EFFECTIVE SURFACE ENERGY, J/m²
150
100
50
0
0
400
800
1200
1600
TEMPERATURE, °C
= STANDARD DEVIATION
TYPE THL Si/SiC


STRENGTH, KSI
60
40
20
0
0
400
800
1200
TEMPERATURE, °C
LONGITUDINAL DIRECTION
TRANSVERSE DIRECTION
= STANDARD DEVIATION

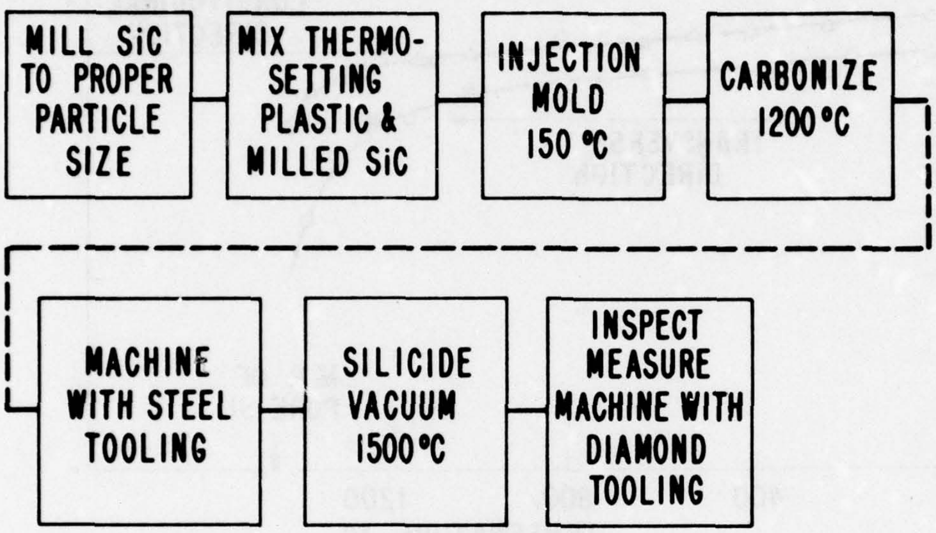


LONGITUDINAL DIRECTION
TRANSVERSE DIRECTION
M.P. OF PURE Si
ELASTIC MODULUS, psi x 10^6
TEMPERATURE, °C
0
20
40
400
800
1200



RATIO MODULUS AT 25°C/MODULUS AT T°C
Si MELTING RANGE
TEMPERATURE, °C
0
0.2
0.4
0.6
0.8
1.0
400
800
1200
1600

INJECTION-MOLDED, REACTION-SINTERED SiC



PROPERTIES OF INJECTION-MOLDED MATERIALS

	Ford SiC	Ford Si_3N_4
Density g/cc	3.14	2.20
Transverse Rupture Strength, Kpsi	55	24
Thermal Expansion 10^{-6}/°C	4.3	1.3
Thermal Conductivity cal/sec-cm-°C	0.50	0.03
E, 10^6 psi	53	17
G, 10^6 psi	22	7

4. CARBIDE-GRAPHITE COMPOSITES*

Transition metal carbide-graphite composites have been thoroughly studied for many years, originally as part of the nuclear rocket engine program and lately as a potentially erosion-resistant nosetip material for ballistic reentry vehicles. The carbides used most recently are those of tantalum and of tantalum-niobium solid solutions. By now, these are no longer considered new materials. Standard operating procedures have been developed for all manufacturing steps, and they are probably better characterized than many commercial materials. As an example, the following list gives the properties that have been determined for mixed NbC·TaC-C composites along the range of property determination. These may be considered vaiable products, well beyond the development stage. A recent reference (Ref. 1) is cited.

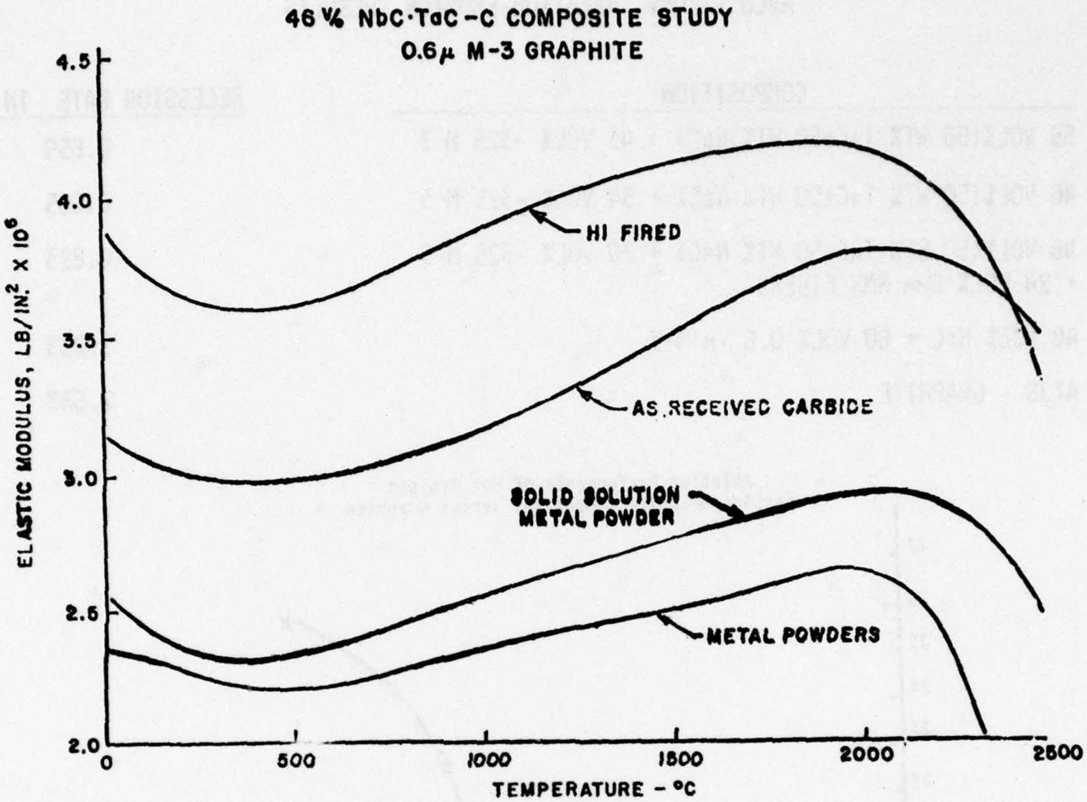
REFERENCE

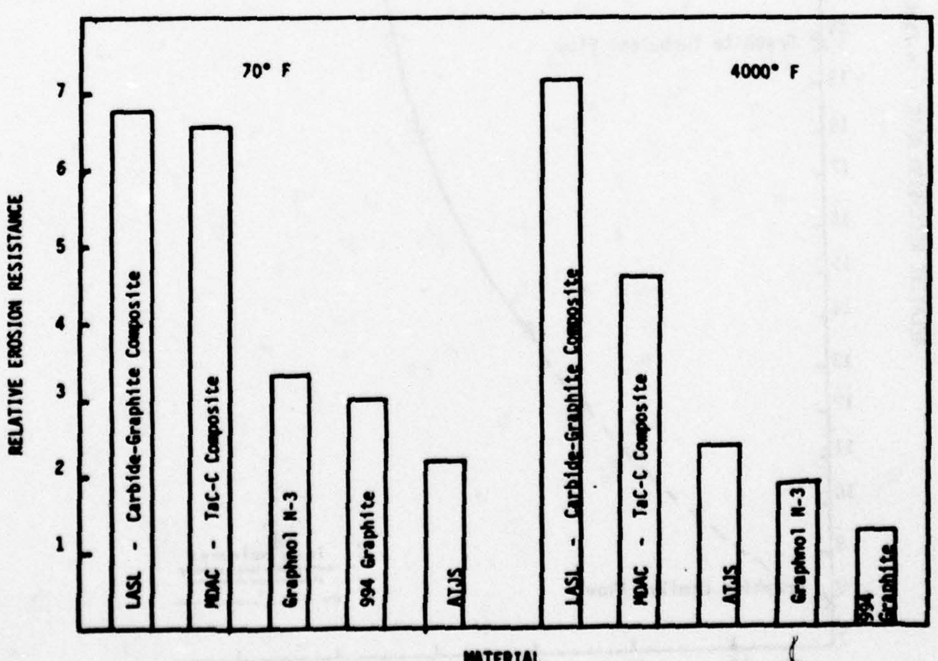
1. C.M. Hollabaugh et al., "Chemical Vapor Deposition of Tantalum on Graphite Cloth for Making Hot-Pressed Fiber-Reinforced Carbide-Graphite Composite," to be published in Proceedings of the Sixth International Conference on Chemical Vapor Deposition, October 10-13, 1977, Atlanta, Georgia.

*Contributor: R.E. Riley.

STUDY OF 46 v/o NbC·TaC-C HOT-PRESSED COMPOSITES
LIST OF PROPERTIES AND EVALUATIONS (Source: Riley)

Property or Evaluation	Conditions or Range	Remarks
Electrical Resistivity	Room Temperature (RT)	Both Parallel and Perpendicular to Pressing Direction
Elastic Modulus	RT to ~ 2500°C	Dynamic Technique, Parallel to Pressing Direction
Thermal Expansion	RT to ~ 2500°C	Optical Tracking, Parallel to Pressing Direction
Thermal Conductivity	Room Temperature	Contact Probe Technique } Parallel Only
	RT to ~ 800°C	Cut-Bar Technique } Parallel Only
Tensile Strength	Room Temperature	Standard Tensile Samples } Parallel Only
Compressive Strength	Room Temperature	Standard Crush Samples } Parallel Only
Bend Strength	RT to ~ 2500°C	1-1/4" Rods, Thirds Point Loading, Parallel and Perpendicular to Pressing Direction.
Notched Tensile Strength	RT to ~ 2200°C	1/4" Dia. Rods with Square Groove, Parallel Only
Compressive Deformation	2700°C, 2000 lb/in^2	30 Minutes } Parallel and Perpendicular to Pressing Direction
	2700°C, 2000 lb/in^2	1 Hour } Parallel and Perpendicular to Pressing Direction
X-Ray	All Pressings	Pressings Showing Non-Uniformities Were Rejected
Chemical and Spectroscopic Analyses	Sample of Each Starting Formulation	Nb, Ta, Total Carbon, Free Carbon; Spec. for Major Impurities (Iron is Major Impurity)
Steady-State Thermal-Stress Resistance	RT to ~ 1500°C	Relative Rankings in Thermal-Stress Fracture Resistance Were Determined on Washer Samples Oriented Perpendicular to the Pressing Direction
Thermal-Shock Thermal-Stress Resistance	RT	Relative Rankings in Thermal-Stress Fracture Resistance Were Determined on Washer Samples Oriented Perpendicular to the Pressing Direction



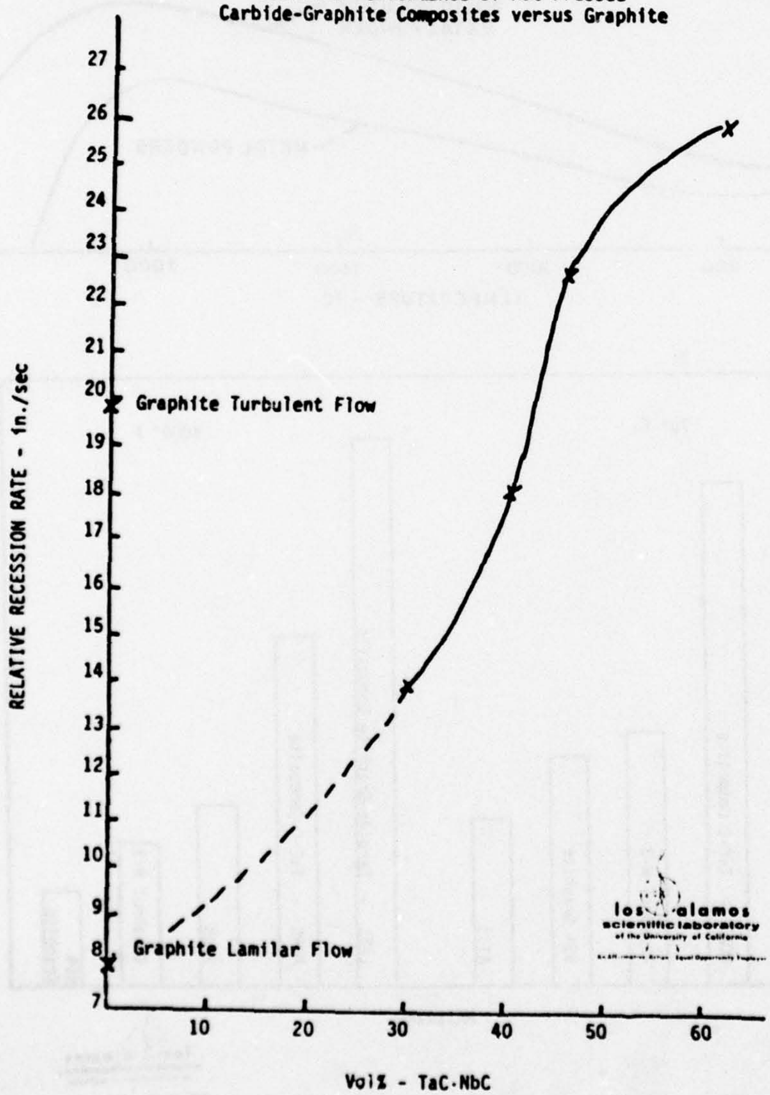


los alamos scientific laboratory of the University of California

AVCO - 10MW ABLATION-EROSION RESULTS

COMPOSITION	RECESSION RATE IN./SEC
55 VOL%(50 WT% TaC+50 WT% NbC) + 45 VOL% -325 M-3	0.659
46 VOL%(50 WT% TaC+50 WT% NbC) + 54 VOL% -325 M-3	0.695
46 VOL%(50 WT% TaC+50 WT% NbC) + 30 VOL% -325 M-3 + 24 VOL% 6mm HMS FIBERS	0.823
40 VOL% HfC + 60 VOL% 0.6 ·m M-3	1.258
ATJS - GRAPHITE	2.587

Ablation Performance of Hot Pressed Carbide-Graphite Composites versus Graphite



5. SILICON NITRIDE MATRIX*

In order to increase the impact and thermal stress properties of silicon nitride, work has proceeded over the last several years to incorporate fibers as a reinforcement. This work is described in a series of United Technologies Research Center reports; the most recent is Ref. 1.

For a variety of reasons brought out in these reports, the reinforcement choice was narrowed down to tantalum wire. In general, such reinforcement gave rise to very large increases in the impact resistance properties and significant increases in resistance to thermal shock. Reinforcement oxidation problems, however, were severe and attempts to presilicide the tantalum did not have much success. The following vugraph-type charts illustrate some of the properties observed in such composites. For similar efforts in Sialons, see Ref. 2.

REFERENCES

1. J.J. Brennan, "Investigate Fiber Reinforced Silicon Nitride," NADC-76147-30, 31 March 1976 (AD A025-901).
2. T. Vasilos, "Development of Fiber Reinforced Sialons," REVMAT Program Final Report on Contract NCD921-75-C-0155 to Avco Corporation by NSWC, 2 April 1976.

*Contributor: J.J. Brennan.

Potential Advantages of Si_3N_4 Matrix Composites

- Increased impact resistance
- Increased strength
- Increased fracture toughness
- Increased thermal conductivity

Candidate Si_3N_4 Matrix Composite Systems

- Continuous fiber reinforcement
  - Ta - Brennan, Rhodes, Vasilos
  - Mo - Brennan, Rhodes, Vasilos
  - Cb - Brennan
  - W - Brennan
  - SiC - Brennan, Lindley
  - C- Brennan, Vasilos
  - Re - Rhodes
- Discontinuous fiber or particle reinforcement
  - SiC - Lange, Richarson, Rhodes, Brennan
  - ZrO_2 - Claussen
  - Si_3N_4 - Rhodes
  - C - Rhodes

Si_3N_4-Ta Impact Properties

Charpy impact

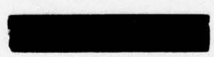
- **Total impact energy for Si_3N_4- Ta increased by a factor of 30 over monolithic Si_3N_4, RT to 1300 °C (15 ft-lbs compared to 0.5 ft-lbs)**
- **Threshold energy for damage for Si_3N_4 -Ta increased by a factor of five over monolithic Si_3N_4, RT to 1300 °C (2.5 ft-lbs compared to 0.5 ft-lbs)**
- **Fracture mode changed so that Si_3N_4 matrix shatters upon impact**

Ballistic impact

- **Energy for ballistic impact damage initiation increased by a factor of 8, RT to 1300 °C (4 ft-lbs vs. 0.5 ft-lbs)**

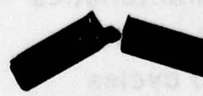
Impact Properties of Si_3N_4 Ceramics

Ta reinforced (3 ft lbs)

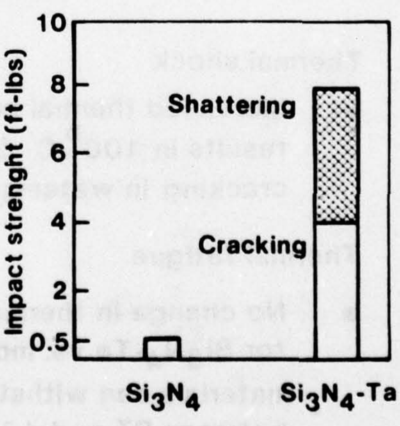


Ta reinforced (18.6 ft lbs)

Monolithic (0.5 ft lbs)



Charpy impact at 75° F



Ballistic impact at 2400° F

Thermal Shock Characteristics of Si_3N_4 and Si_3N_4 -Ta Composites

Test - Heating test samples in air to temperatures from 400^{o} C to 1400^{o} C followed by quenching in cold water.

Results - Si_3N_4 plate of 1/8in x 5/8in x 1 1/4 in dimensions withstood a ΔT of up to 600^{o} C with little or no damage or loss in properties, using either 5% MgO or 10% Y_2O_3 additives. Initial test on Si_3N_4-Ta plates showed that a ΔT of greater than 600^{o} C was necessary to cause damage

Si_3N_4-Ta Thermal Shock and Fatigue Properties

Thermal shock

- Increased thermal conductivity for Si_3N_4-Ta results in 100^{o} C ΔT increase to cause cracking in water quench thermal shock tests

Thermal fatigue

- No change in thermal fatigue characteristics for Si_3N_4-Ta vs. monolithic Si_3N_4. Both materials can withstand over 600 cycles between RT and 1300 ^{o}C in fluidized bed tests.

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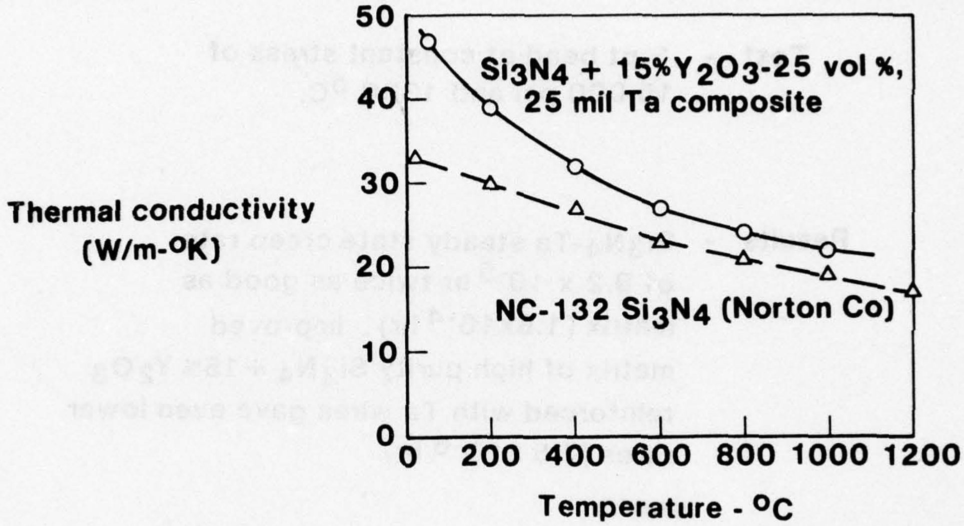
Thermal Conductivity vs Temperature for Ta Reinforced Si_3N_4 and Norton NC-132 Si_3N_4



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Si_3N_4-Ta Fracture Mechanics

Notched beam tests

RT critical stress intensity factor (K_{IC}) of Si_3N_4-Ta higher than matrix alone. (8.57 $MN/m^{3/2}$ vs 6.75 $MN/m^{3/2}$).
Si_3N_4 -Ta K_{IC} at 1300 °C lower than matrix alone. (5.36 $MN/m^{3/2}$ vs. 8.00 $MN/m^{3/2}$)

Double torsion tests

Crack growth rates in Si_3N_4-Ta comparable to matrix (Y_2O_3 additive) at RT and 1300 °C.

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Si_3N_4-Ta Creep Properties

Test - 3-pt bend at constant stress of 15,000 psi and 1300 °C.

Results - Si_3N_4-Ta steady state creep rate of 9.2×10^{-5} hr twice as good as matrix (1.8×10^{-4} hr). Improved matrix of high purity Si_3N_4 + 15% Y_2O_3 reinforced with Ta wires gave even lower rates (1.5×10^{-6} hr).

Si_3N_4-Ta Liabilities

Decrease in strength

	RT MOR	1300 °C MOR	RT Tensile
Si_3N_4 – 15% Y_2O_3	130 ksi	100 ksi	85 ksi
Si_3N_4 + 15% Y_2O_3 - 25% Ta	100 ksi	70 ksi	25 ksi

Increase in density

Si_3N_4 + 15% Y_2O_3 - 3.4 gms/cc

Si_3N_4 + 15% Y_2O_3 + 25 vol% Ta - 6.7 gms/cc

Decreased oxidation resistance

Increased fabrication difficulty

6. FILAMENT REINFORCED GLASS-MATRIX COMPOSITES*

The reinforcement of glass matrices with advanced filaments dates back to the late 1960-early 1970 period when both boron and carbon fibers were utilized successfully to form high strength composites. At that time, however, the much greater emphasis on resin and metal-matrix composites tended to retard progress in this area. The recent resurgence of interest in ceramics and intermetallic compounds as structural materials, as well as the great interest in achieving composite materials with higher use temperatures, has warranted a reexamination and extension of glass-matrix composite technology.

Four continuous high-performance filaments are currently being used to reinforce glass-matrix composites: two large diameter monofilaments (boron and silicon carbide) and two multi-filament yarns (carbon and alumina). It can be shown that the use of each of these fibers provides unique advantages and, in some cases, disadvantages to the resultant composite.

The mechanical properties of these systems are discussed and it is shown that very high levels of axial tensile strength can be achieved over a wide range of temperature. These strength levels exceed those of any currently available composites at temperatures above 400°C on both an absolute and specific (strength divided by density) basis. Of even greater possible significance is the demonstration of composite fracture toughness equivalent to that of metallic engineering alloys.

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*Contributor: K.M. Prewo.

At present, it is perhaps too early to predict the future of glass-matrix composite applications. The current interests in alumina fiber reinforced glass as a radome material, and graphite reinforced glass for high-temperature structures, are still only emerging. It will require significantly more interest and time on the part of designers and materials scientists to find the unique combination of material and application that will lead to the first engineering use. This is, however, as it has always been for new composite systems.

Previous Work

- Boron reinforced glass
  - Siefert 1971-1974
- Carbon reinforced glass
  - Sambell and Phillips 1972-1974
- Carbon reinforced lithium aluminosilicate
  - Levitt 1973

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Why Look at Glass Matrix Composites Again?

- New and lower cost fibers
- Advanced status of composite material technology
- Resurgence of interest in ceramics
- Interest in use of intermetallic compounds (TiAl, Ti_3Al)

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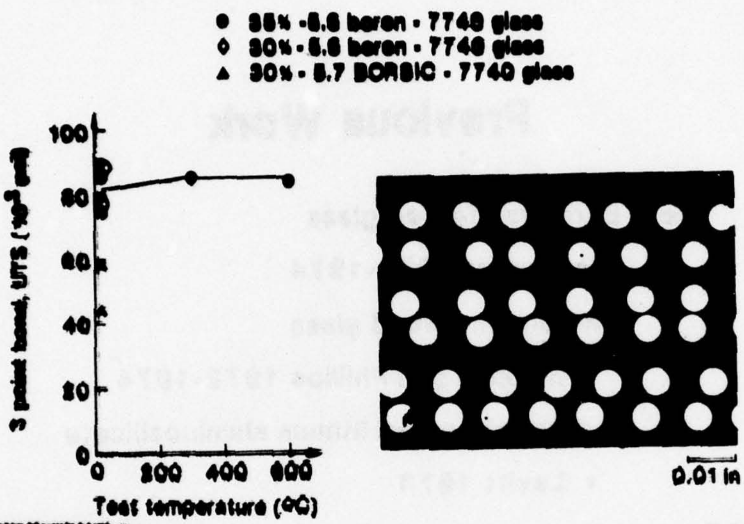
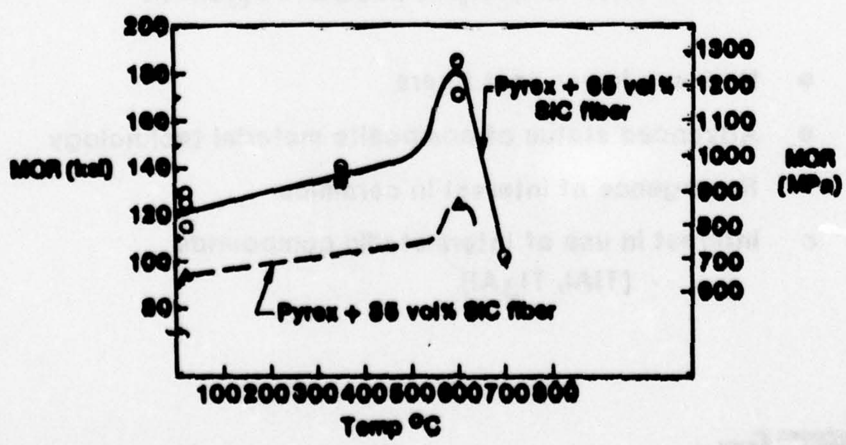
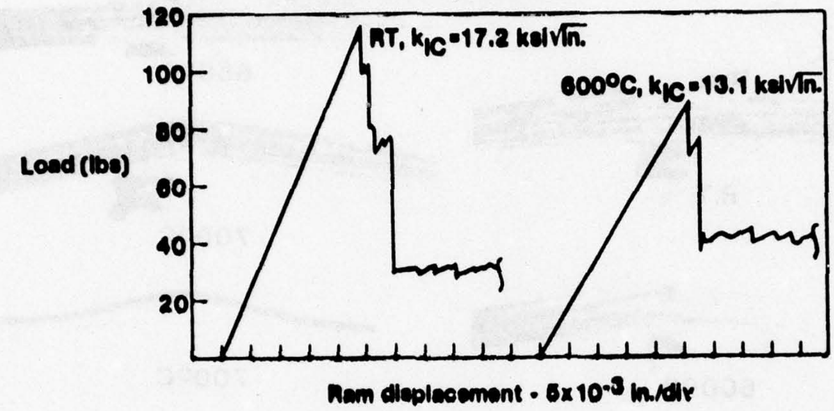
BORON REINFORCED GLASS



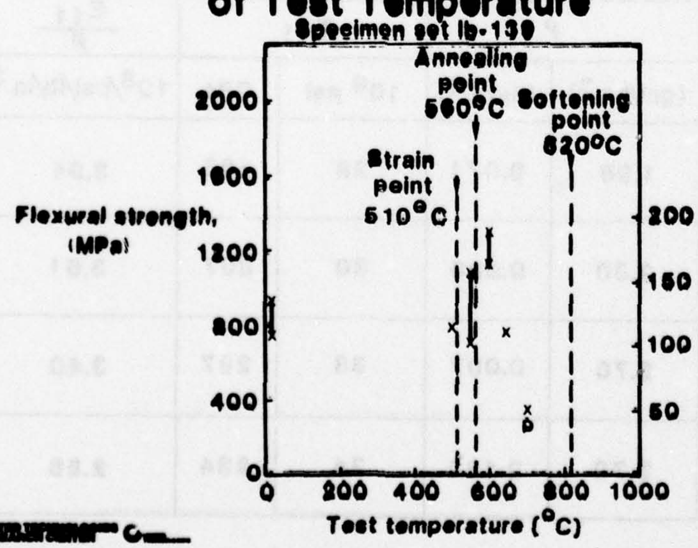
3-pt Bend Strength in Air vs Temperature for Pyrex 7740 Glass - 5.6 Mil SiC Composite



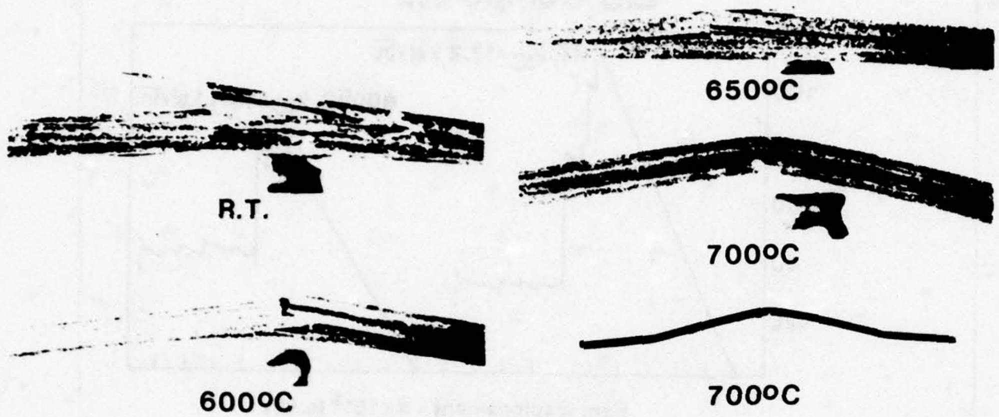
Notched Beam Load vs Displacement Curves for Pyrex 7740 Glass/35 vol%, 5.6 Mil SiC Composite



Flexural Strength of Hercules HMS-10K Fiber Reinforced 7740 Glass as a Function of Test Temperature

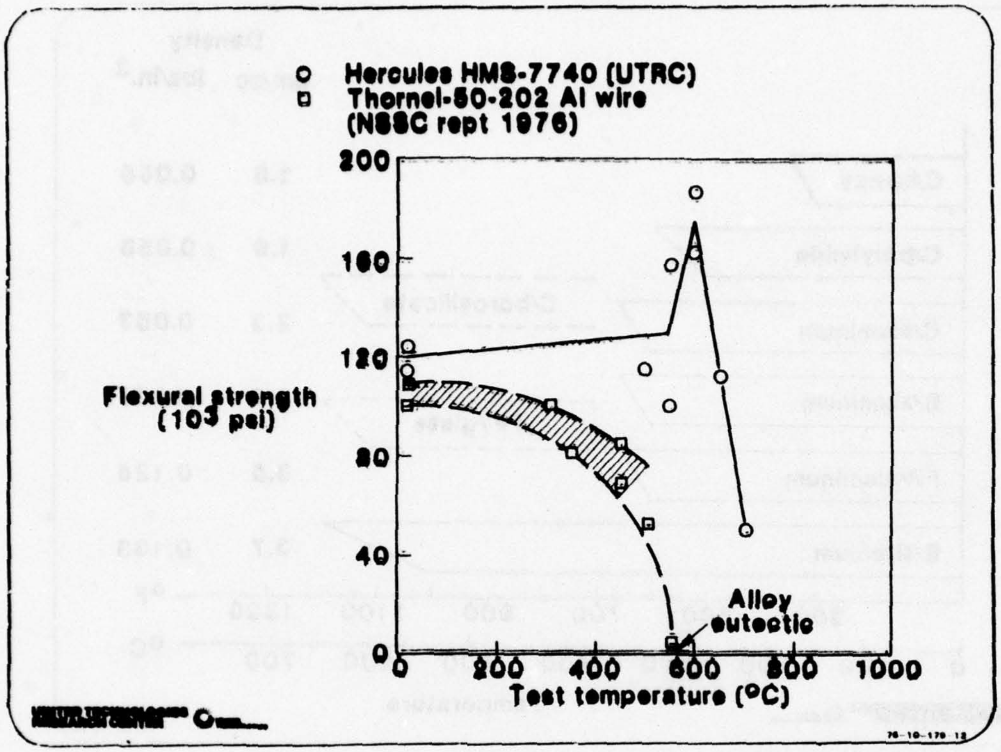


HMS-7740
Three Point Flexural Test



Elastic Modulus Comparison

System	ρ		E_{11}		$\frac{E_{11}}{\rho}$
	(gm/cm^3)	(lb/in.^{3})	10^6 psi	GPa	10^8/psi/lb/in.^{3}
50 v/o HMS-7740	1.96	0.071	28	193	3.94
42 v/o T50-Al	2.30	0.083	30	207	3.61
50 v/o B-Al	2.70	0.097	33	227	3.40
43 v/o B-Ti	3.70	0.133	34	234	2.56

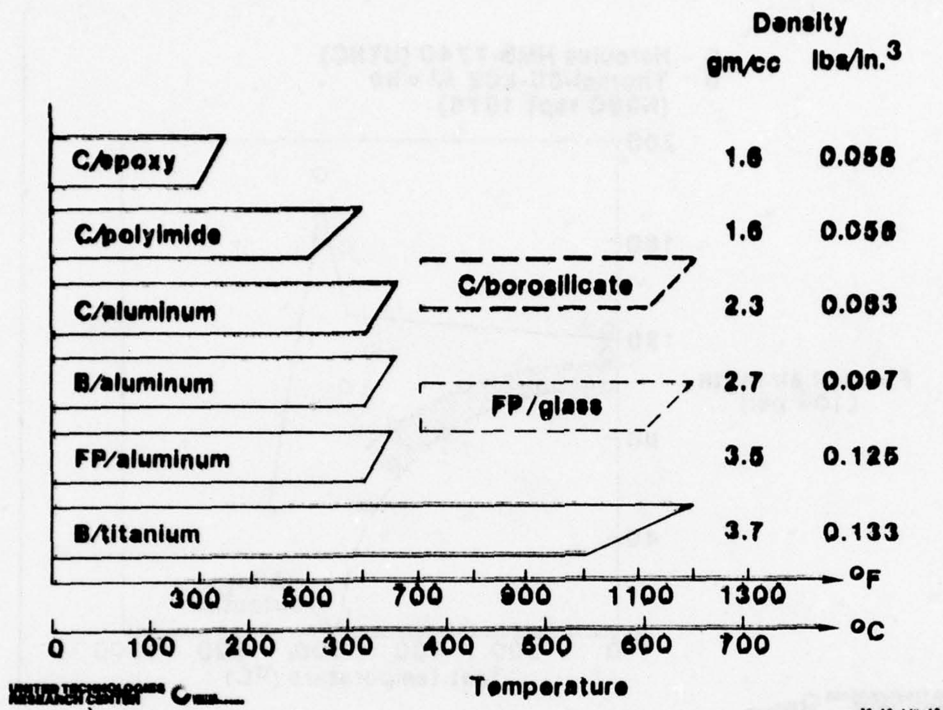


Fracture Toughness Specimen Test Data

HMS- 7740

Specimen	Test Speed (cm/min)	Test Temp °C	K_c $MN/m^{3/2}$	K_c $10^3 psi\sqrt{in}$	Energy per unit area Joules/m^2	Energy per unit area ft - lbs/in^2
LB-140-1	20,000	22	21.4	19.5	23,500	11.3
-2	0.127	22	22.1	20.1	–	
LB-157-1	20,000	600	15.8	14.3	10,600	5.1
-2	20,000	650	19.0	17.3	11,800	5.7

77-03-208-5



Future of Glass Matrix Composites

- **Advantages**
  - High 0° UTS
  - Higher use temperature
  - Environmental stability
  - Low density
  - Low cost materials
- **Limitations**
  - Low 90° UTS
  - Low failure strain
  - Fabrication conditions

7. GENERAL TECHNOLOGY OF CERAMIC COMPOSITES*

A general review is given of the emerging concepts of reinforcement toughening of ceramics together with some historical background. Criteria are given as guidelines in modeling such composites. One major reason for prior unsatisfactory efforts to fabricate composites has been the use of large-size particles or fibers. A general rule of thumb is that this size must be significantly smaller than the critical flaw sizes. Some of the present theories of reinforcement toughening are given and some supporting data.

A short discussion of fabrication techniques is also given. Two concepts that appear attractive are sol-gel processing (where possible) and polymer pyrolysis. For the latter process (forming ceramic fibers *in situ* by pyrolyzing organic fibers), copyrolysis is possible and offers some potential flexibility in incorporating different kinds of fibers into the same matrix.

While the following vugraphs are not annotated, they are self-explanatory.

---

*Contributor: R.W. Rice.

CONCEPTS FOR "TOUGH" CERAMIC COMPOSITES

*OLDER:

1) Crack stoppers: Pores, Particles, Fibers
2) Reduce E or $\frac{\sigma^2}{2E}$
3) Wire or fiber reinforcement

*NEWER:

	Mechanism Reinforcement	Energy Absorption	Toughening at: R.T.	H. T.
1) Fine fiber toughening	x	x	H	M
2) Fiberous pullout		x	M	M?
3) Phase transformation		x	H	L
4) Line tension		x		L
5) Microcracking		x	H	L
6) Dispersion strengthening	x		L	H

*Not to be confused with second phase control of microstructure.

Wire Reinforced Ceramics*

1317

Ballistically Tested Alumina Specimen

1426

Ballistically Tested Alumina-Nichrome Wire Mesh Composite, 3 16x16/in Wire Meshes

Ballistically Tested Alumina-Nichrome Wire Mesh Composite, 3 8x8/in Meshes

*After Johnson & Morgan
AFML-TR-70-54, Part II

Earlier Ceramic Particulate Composites*

Hypereutectic carbide

BeO-10 v/o BN

*After Rossi, Aerospace Corp.

$$\sigma = A\sqrt{\frac{E_c \gamma}{c}}$$

$E_f > E_m$ increase E_c, increases σ

1) Modulus limitation

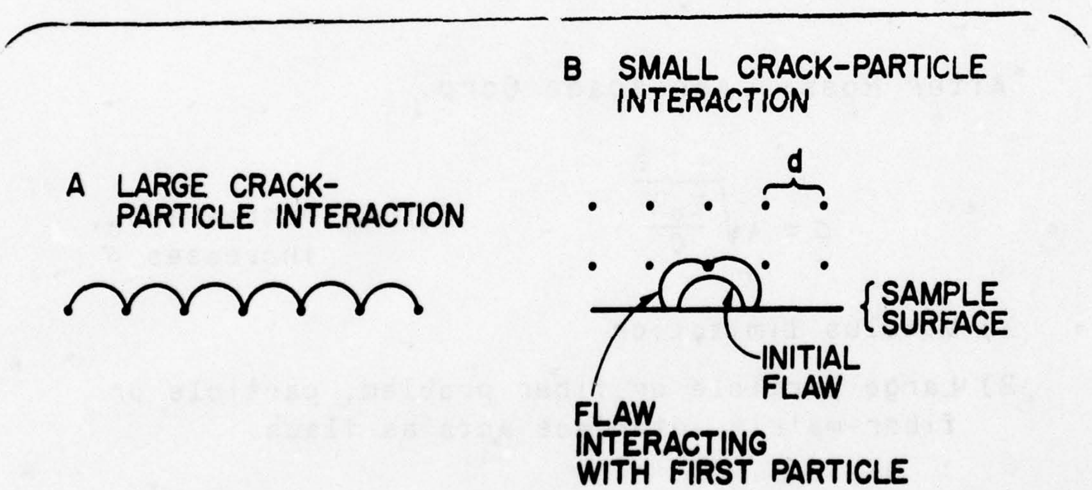
2) *Large particle or fiber problem, particle or fiber-matrix interface acts as flaws*

Fiber	Process	Typical Diameter(μm)
Metal	Drawn	25-200
Glass, Al_2O_3	Melt drawn	50-250
B, SiC, B_4C, TiB_2	CVD	50-300
C, Al_2O_3, SiC	Pyrolysis Sintering	5-20

Common ceramic flaw sizes 20-50μ

LIMITATIONS OF OLDER CERAMIC COMPOSITES

1. Modulus limitations.
2. Large particles or wires acted as flaws - low strength, limited toughness increase.
3. Matrix flaws controlling.
4. Lack of synergism between matrix and fibers.
5. Lack of oxidation resistant fibers.



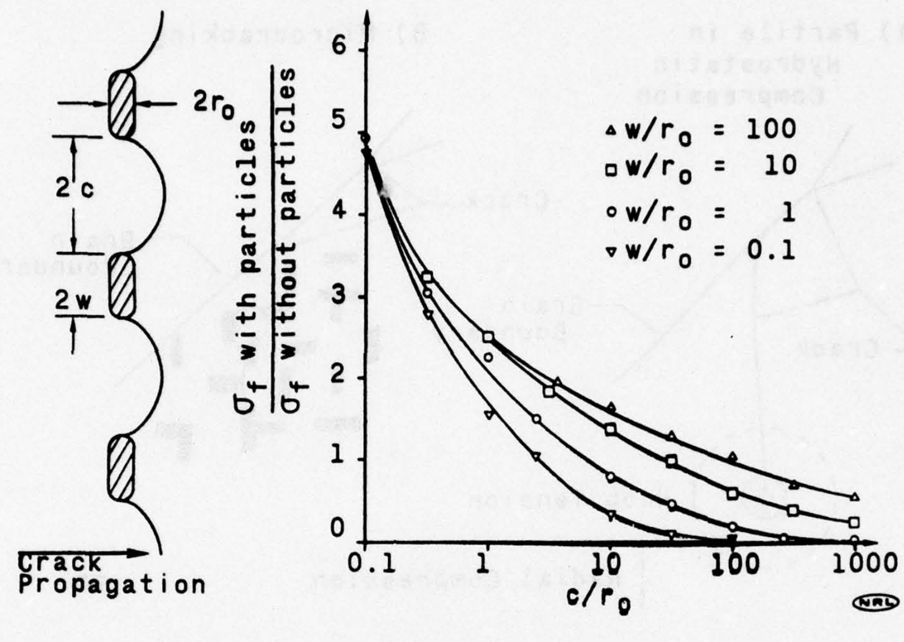
$$\gamma = \gamma_0 + \frac{T}{d} \qquad T = \frac{2r\gamma_0}{3}$$

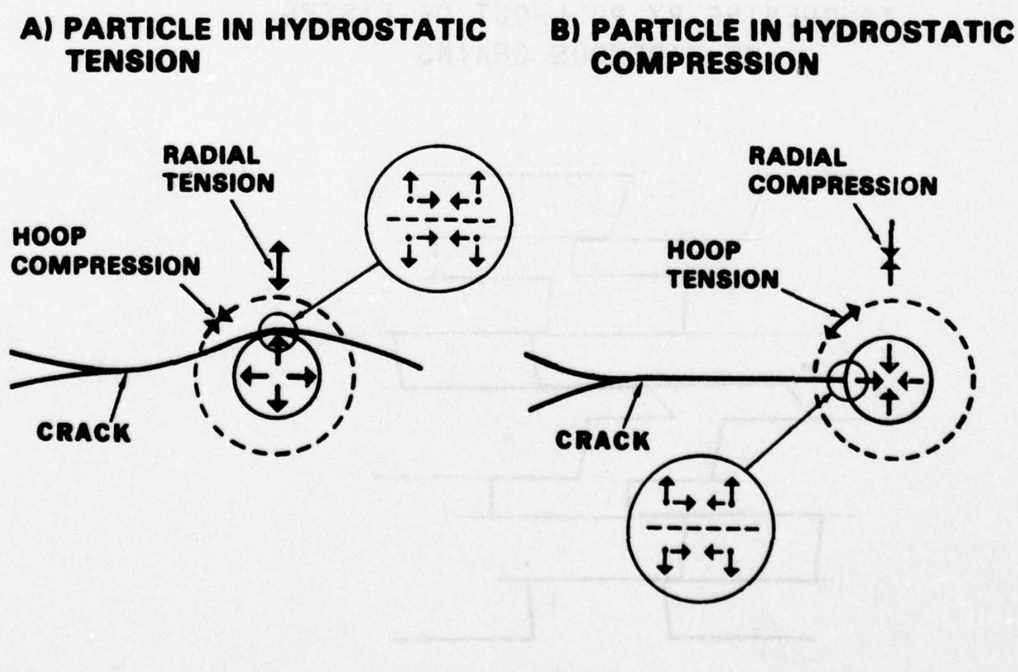
r = FLAW RADIUS

γ_0 = MATRIX FRACTURE ENERGY

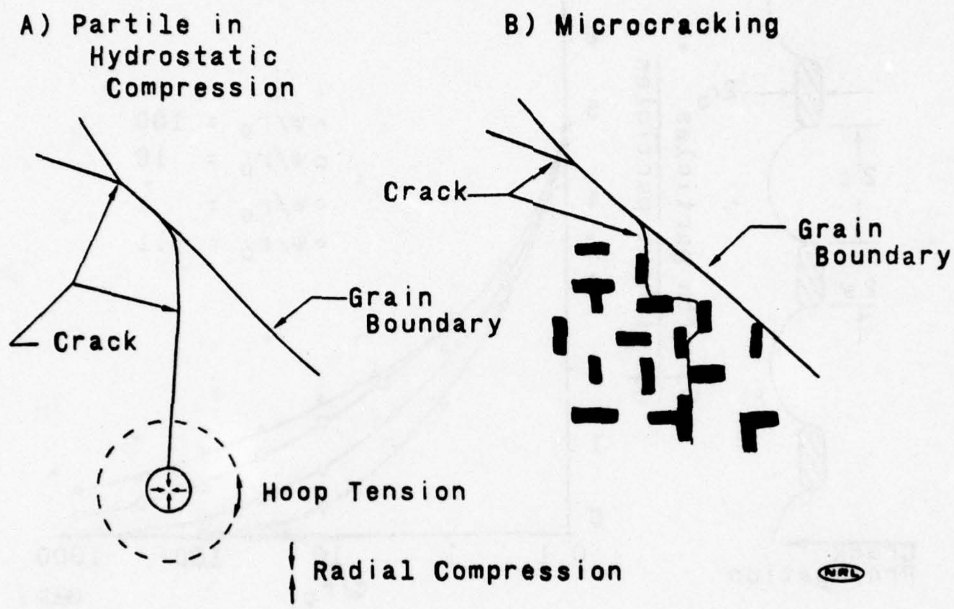
d = PARTICLE SPACING

LINE TENSION STRENGTHENING

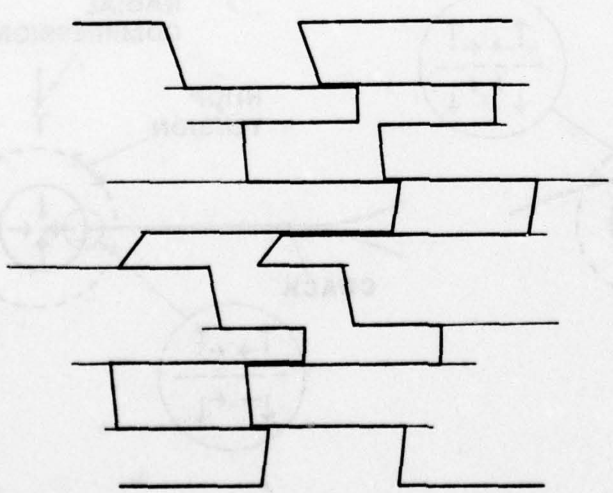




AVOIDING INTERGRANULAR FAILURE
(in Large Grain Ceramic Composites)



TOUGHENING BY PULL-OUT OF FIBERS
OR FIBEROUS GRAINS



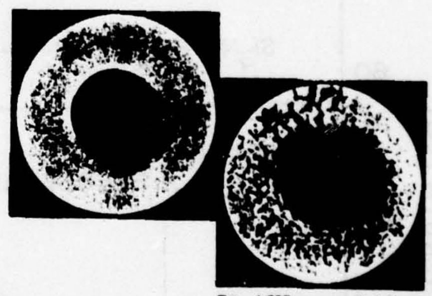
CRITERIA FOR CERAMIC PARTICLE COMPOSITES

1) Small particle size
2) Medium-high particle density (small particles)
3) Particle spacing > particle size
4) Control microcracking

$$D \geq \frac{12}{(\Delta \epsilon)^2} \frac{\gamma}{E}$$

PARTIALLY STABILIZED ZrO_2 (PSZ)

	Strength 1000 psi	Fracture Energy J/m^2
Large Grain PSZ	60-90	250
Fine Grain PSZ	80-150	100
H.P. Si_3N_4	100-150	25-125
Typical Al_2O_3	50	20-40

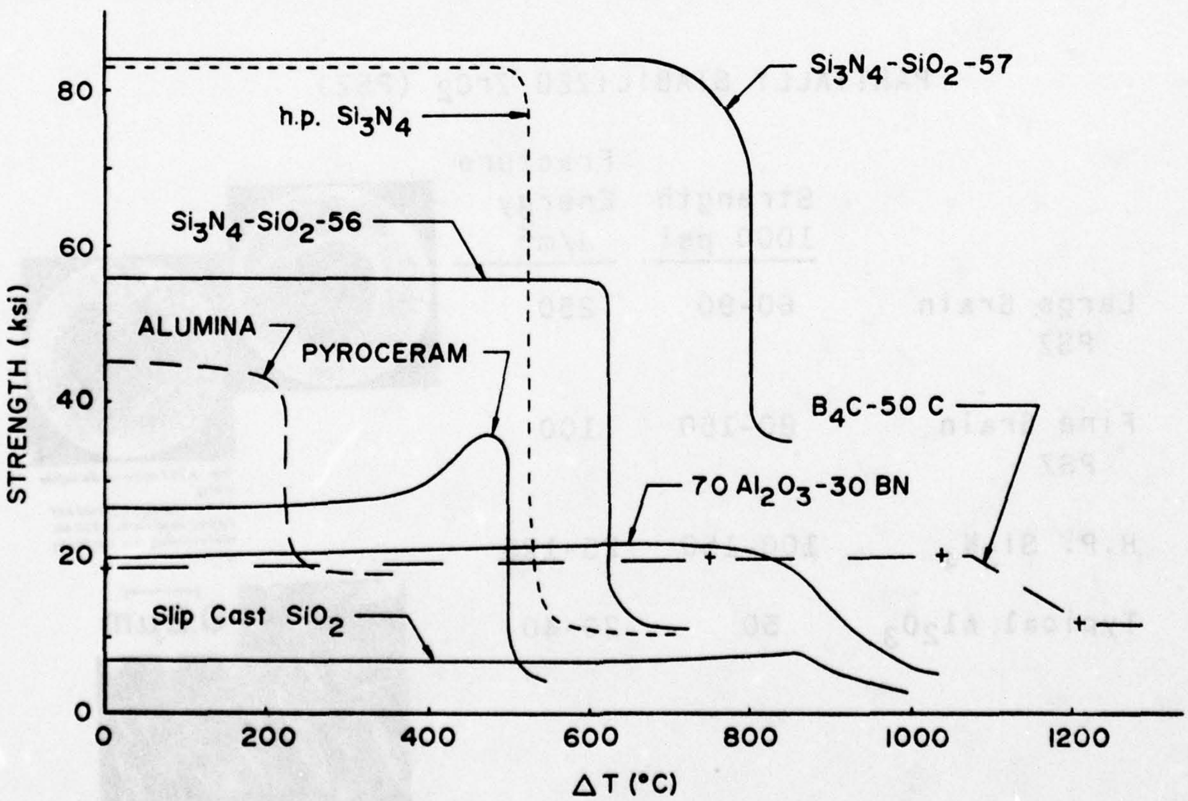


Top: A PSZ extrusion die before testing
Above: Slightly roughened and showing small cracks, a PSZ die after extruding over 100 000 kg of brass

CERAMIC COMPOSITES

Composition	Approx. Porosity %	E 10^6 psi	K_{IC} $MN/m^{3/2}$ ⊥	K_{IC} $MN/m^{3/2}$ ∥	σ_f (ksi) ⊥	Thermal Shock Resistance Quench	Thermal Shock Resistance Laser
Al_2O_3-30 v/o BN	4	28	4-4.5	3	20-30	Exc/850	Exc.
Al_2O_3-50 v/o BN	11	30	2.3	1.3	12-15	Exc/100	Exc.
Al_2O_3-30 v/o CSZ	4		3.9		40-50	Poor/200	
Al_2O_3-30 v/o USZ	4		3.9		10-30	Poor/250 Fair/400	Good
B_4C-50 v/o C + 5 v/o Al_2O_3	5				20-30	Fair/600 Exc/1000	Exc.

COMPARATIVE THERMAL SHOCK BEHAVIOR



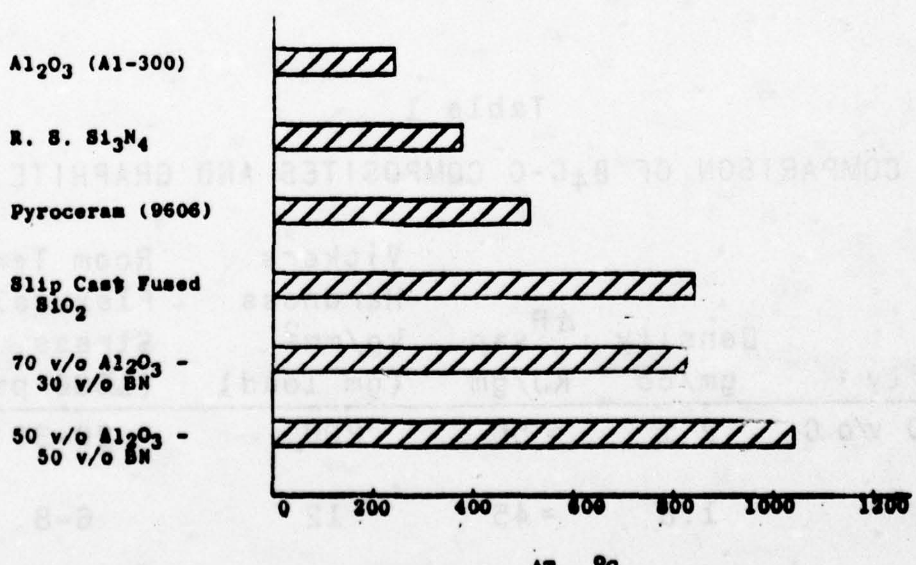
DIELECTRIC PROPERTIES OF CANDIDATE RADOME MATERIALS

Material	Density g/cc	Dielectric Constant at 8-10 GHz	Loss Tangent
Slip Cast Fused Silica	2.6	3.3	0.4
Pyroceram 9606	2.45	5.65	0.2
Si_3N_4 (reaction sintered)		5.6	2.0
Al_2O_3	2.9	9.5	0.1
Iso. pressed BN	2.5	3.2	0.1
Hot pressed $50Al_2O_3$-50BN	2.8	6.5	0.4

*Data from Bull. Am. Ceram. Soc. 53 (3), 255, 1974 (J. D. Walton, Jr.) except for Al_2O_3-BN composites which were measured at MIT.

NRL

CONVENTIONAL THERMAL SHOCK RESISTANCE OF RADOME MATERIALS



ΔT_c: Temperature from which sample is rapidly quenched (in H_2O) to 25°C to cause loss in strength at 25°C.

COMPARATIVE RAIN EROSION RESISTANCE OF RADOME* MATERIALS

Material	Time to Damage	Condition of Samples
Pyroceram 9606 Fortified	10 min.	Limited erosion
Alumina	5 min.	Limited erosion
70 Al_2O_3 - 30 BN	1 min. 35 sec.	Considerable erosion, failure through delamination/cracking
60 Al_2O_3 - 40 BN	1 min.	Cracks formed
50 Al_2O_3 - 50 BN	30 sec.- 1 min.	Cracks formed
Slip Cast Fused Silica	40 sec.	Cracks formed

* Run at Bell Aerospace Division of Textron
Rain Field: 1 in/hr (90^o to rain)
1.8-2 mm Avg. drop size
Velocity - 732 ft/sec
20 in. vacuum

Table 1

COMPARISON OF B_4C-C COMPOSITES AND GRAPHITE

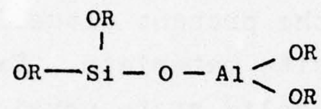
Property	Density gm/cc	ΔH_{vap} KJ/gm	Vickers Hardness kg/mm^2 (gm load)	Room Temp. Flexural Stress (1000 psi
B_4C-50 v/o C	2.1	≈ 50	200	20-30
ATJS	1.8	≈ 45	12	6-8

SOL-GEL PROCESSING

$$\text{SOL (e.g. AlO(OH) SOLUTION)} \xrightarrow{\text{DRY}} \text{GEL} \xrightarrow{\text{FORM}} \text{PYROLYSIS} \longrightarrow \text{CERAMIC: e.g. } Al_2O_3$$

ADVANTAGES:

1) PURE MATERIALS: STARTING MATERIALS CAN BE FILTERED, DISTILLED, "TEST TUBE" ENVIRONMENT PREVENTS CONTAMINATION.
2) HIGHLY REACTIVE $\longrightarrow$ LOWER SINTERING TEMPERATURES.
3) INSPECTION - TRANSPARENCY.
4) MOLECULAR MIXING IN SOL FOR COMPOSITES MIXED COMPOUNDS AND DOPANTS,

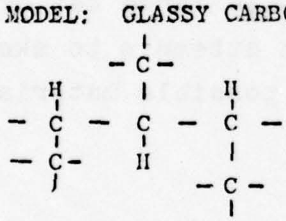
   e.g.: 

5) SOLID-SOL MIXING-COMPOSITES

CERAMICS FROM POLYMER PYROLYSIS

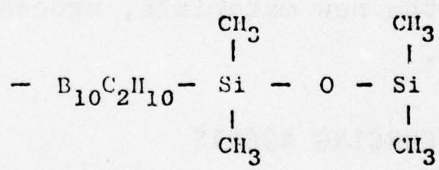
$$\text{SHAPED POLYMER} \xrightarrow{\Delta} \text{SHAPED CERAMIC}$$

MODEL: GLASSY CARBON



INITIAL APPLICATION:

$B_4C + SiC$:



INTERACTIONS: K. WYNN (ONR), W. FOX AND P. NORDQUIST, NRL

UNIQUENESS: FINE PORE SIZE, UNIQUE MICROSTRUCTURE, COMPLEX SHAPES, PROPERTIES

ADVANTAGES: PURITY, SHAPING, LOW TEMPERATURE, FREEDOM FROM ADDITIVES, IN SITU COMPOSITES

APPLICATIONS: COMPOSITES, FIBERS, ELECTRONIC MATERIALS

CERAMIC COMPOSITES

Future

Promising

Needs

1. Property prediction of phases
2. Understanding mechanisms
3. Phase data
4. Processing

8. INNOVATIONS IN CERAMIC COMPOSITE FABRICATION TECHNIQUES AND ASSOCIATED TECHNOLOGIES*

The application of existing innovations to new fields is defined as technology transfer. This phenomenon most appropriately characterizes the present situation in the field of reinforced ceramic composite materials. Expertise from polymer synthesis chemistry, solid-state physics and mechanics, gas-dynamics, thermodynamics, and kinetics are being brought to bear upon an area once populated only by ceramists.

New developments in reinforcing agents and matrices, when combined with a variety of processing and fabrication techniques potentially available, result in an extremely large set of composite variants. The following discussion attempts to sketch some of the new materials, processes, and possible material variants.

A. REINFORCING AGENTS

Conventional reinforcements most commonly conceived are multiple filament, continuous fiber bundles including carbon, metal oxide, nitride and carbide fibers, superally and refractory metal wires. Specific examples would include carbon fibers of polyacrylonitrile, rayon and pitch heritage, alumnia fiber, boron nitride fibers, silicon carbide fibers, MAR-M509 wire and tungsten wire.

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*Contributors: E.L. Paquette, H. Herman (who did not attend the workshop) and C.F. Cline (whose material was not available for inclusion).

In addition to these reinforcements, *in situ* whiskers, such as those reported by Hulse (Ref. 1), and ribbon reinforcements, such as the carbon ribbons developed by Froberg (Ref. 2), demonstrate the potential for alternative refinforcements whose properties may be better suited for a specific application.

Current high-performance polymer composites now utilize blends of fibers including high-performance carbon and such fibers as Kevlar or S-glass. As the level of sophistication increases in the field under discussion, similar engineering or economic optimization may occur, particularly if destructive interactive phenomena can be contained, if indeed they are present.

The engineering possibilities resulting from this approach are multiple, such as increasing the uniformity of composite response to changing environmental factors (temperature, cyclical or static stress, impact and thermal shock).

The heterogeneous nature of the composite allows isotropic materials to produce anisotropic composite properties. Geometry-dependent thermal expansion and conductivity phenemona are frequently observed.

B. MATRICES AND PROCESSES

Matrices may be consolidated through the use of powders and solid-state sintering, gas-solid diffusional reactions, gaseous infiltration and deposition or liquid impregnation and *in situ* pyrolysis.

Current work by Congdon (Ref. 3) on a pressure slip cast alumina/alumina composite (Sapphil fibers and Linde A alumina matrix) demonstrates increased K_{IC} for short fibers at 10 to 15 volume percent, despite lowered densities and flexural strength.

Mazdeyasni and West (Ref. 4) will report on liquid infiltration and pyrolysis process research using high molecular weight silicon-containing organic polymers. Significant improvements in properties of porous reaction sintered silicon

carbide bodies will be reported by them at the September 25-28, 1977 meeting of the American Ceramic Society at Hyannis, Massachusetts.

Yajima et al. (Ref. 5) have reported on the development of SiC fibers from a polycarbosilane precursor (M.W. $\simeq$ 1200) via pyrolysis in vacuum. This same class of metal-containing polymer may be used, as Mazdeyasni and West have done, to fill the porosity of a porous sintered ceramic body or a low density composite.

Gaseous matrix fabrication techniques include physical and chemical vapor deposition (CVD); plasma spraying or sputtering are also potential deposition techniques applicable to thin laminates. Chemical vapor deposition has been applied to the surface of many monolithic ceramics such as reaction-bonded Si_3N_4 to increase oxidation protection. CVD has also been used to rigidize and densify fibrous preforms.

As in carbon-carbon technology, fiber-matrix interfaces will be critically important in terms of load translation, interface corrosion phenomena between dissimilar materials, fatigue, and crack propagation. The gaseous techniques, in particular, are generally the process vehicle of choice when diffusion barriers are indicated. In addition, the current generation of ceramic fibers is very abrasion-sensitive due to the generally anisotropic nature of the filament cross section resulting in the sheath or filament perimeter representing most of the developed fiber strength. Abrasive processing damage could be minimized in the first generation of composites by avoiding powder-based matrix materials.

Riley and Wallace (Ref. 6) will report at the Sixth International Chemical Vapor Deposition Conference in October [1977] on the fabrication of a tantalum carbide-carbon composite formed by tantalizing graphite fibers via CVD and subsequently hot-pressing to form a tantalum carbide-matrix-carbon fiber core

composite. It would be interesting to speculate on the results attainable if hot isostatic pressing was applied to this composite or to the pyrolysis of a polycarbosilane-containing composite.

Economy, Smith, and Lin (Ref. 7) reported fabrication of a B_4C fiber-carbon matrix composite using six micron B_4C fibers and furfuryl alcohol resin-derived carbon matrix with a CVD SiC coating. The composite demonstrated interlaminar shear strength of over 7000 psi and had negligible weight change (due to oxidation) when statically held in 1000°C air for over 30 hours. Replacement of the carbon phase with silicon carbide, which has a nearly ~~ident~~ical thermal expansion curve, could lead to a reliable oxidation-resistant long-life composite.

In the longer term, more sophisticated ceramic composites may use several fabrication processes and possibly even dissimilar matrix materials as previously mentioned about blended reinforcements.

The use of chemical vapor deposition to rigidize a fibrous ceramic body prior to liquid phase impregnation and pyrolysis immediately comes to our attention. Other sequences or choices of materials may offer processing, economic, or applications incentives.

In an effort to maximize the data obtainable for experimentally fabricated composites and readily assess their performance potential, Bird, Newquist, and Paquette (Ref. 8) will report at the American Ceramic Society Conference at Hyannis, Massachusetts, on predicting properties of ceramic composites using a micromechanics model originally developed for carbon-carbon composites.

While plasma spraying (and more generally, thermal spraying) are thought of as coating techniques, it is indeed possible to fabricate a free-standing form using this method. The melting of oxides and other refractory ceramics is easily accomplished in this method, the spraying parameters being readily controllable

(e.g., gas type and flow rate, power, particle size). A limitation which must always be recognized is the inherent porosity that will result from spraying. This, however, can be mitigated somewhat by spraying in a controlled atmosphere or vacuum and by a judicious choice of particle size. This problem of porosity will not easily be made to go away and the best approach may be to consider post-spraying sintering, pore filling or, perhaps, laser glazing to seal the surface of the structure. Hot isostatic pressing may be appropriate for certain high-performance products having the proper shapes.

For a moment, let us consider the benefits of plasma spraying. A wide range of ceramic materials has been sprayed as coatings. The major limitation is that the material not decompose below its melting point. Obviously, most oxides and carbides are sprayable, and large numbers of composite coatings have been formed. Some are self-sealing due to extra heat-of-reaction processes occurring on solidification. The greatest amount of experience has been obtained with Al_2O_3, with an without ZrO_2, Y_2O_3, etc. There are a large number of potential applications [cathode-ray tube (CRT), for example] where both coatings and bulk products of spray-formed Al_2O_3-Y_2O_3 could be used. The yttria act as a glass former, giving rise to a glass-ceramic system with improved properties. We have found that TiO_2 added to Al_2O_3 has a similar effect, but to a lesser degree.

There are too many oxide and carbide systems to consider here, but it should be noted that under proper control a composite could be formed with a precipitation-strengthened matrix. Plasma spraying yields highly metastable states, and extreme supersaturation with subsequent heating could be used to create, for example, spinodal decomposition, ultrafine glass-ceramic, etc.

Powder preparation is a significant issue affecting plasma spraying, because the melting, transport properties, and solidification processes depend importantly on particle size. Thus, one may mix and spray two particle types that differ greatly in size, and obtain an interesting composite, Particle size and,

to an extent, particle shape must be considered in the larger context of controllable spray parameters. There are possibilities, also, for decreased expense through the use of naturally occurring minerals.

The mechanical properties of ceramics that have been formed by plasma spraying have received limited attention. As a facing, the plasma-sprayed oxide or carbide works well. The pores are frequently sealed by the plasma torch, but laser glazing could be used more effectively in a controlled manner. In the free-standing form, consideration must be made of the occurrence of microcracking. The literature now contains numerous reports on this matter, especially as how it affects thermal stress resistance. Microcracking can lead to an increased strain-to-fracture, which is what one generaly is attempting to do with ceramics. Plasma-sprayed bodies, almost by definition, contain a large number of microcracks. These structures (at least in the coating form) are stronger than would be expected. It may be thus that the improved strength characteristics of an Al_2O_3-ArO_2 plasma-sprayed coating may be due to process-induced microcracks. The impact and erosion resistance of plasma-sprayed free-standing forms may then be improved by the very presence of the micro-defects.

There are a number of European organizations currently interested in free-standing bulk forms created by plasma spraying. Harwell in the United Kingdom is developing these products of Al_2O_3 and ZrO_2, and these are claimed to be of high density. FIAT has recently developed an interest in lazer-glazing plasma-sprayed products, and a member of the staff of their research laboratories indicated that he is interested in applying this process to free-standing forms also. Aluswiss in Zurich has created an entire line of free-standing form, but no work is apparent on ceramic-matrix composites formed in this manner.

In summary, existing processes and technologies adapted or transferred to the field of ceramic composites can reduce the development time scale and attendant expense to a fraction of the effort expended to date on carbon and boron fiber-derived composite families.

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2. R.W. Froberg and W.A. Robba, Method of Making a Film of Refractory Material Having Bidirectional Reinforcing Properties, U.S. Patent 3,900,540.

3. J.W. Congdon, current Ph.D. thesis under tutelege of Dr. David Lewis, III, Alfred University, Michigan.

4. K.S. Mazdeyasni, AFML, Wright-Patterson AFB, Ohio and R. West, University of Wisconsin, "Characterization of Organosilicon Infiltrated Porous Reaction Sintered SiC," to be presented at the Basic Science and Nuclear Division Joint Fall Meeting, American Ceramic Society, September 25-28, 1977, Hyannis, Massachusetts.

5. S. Yajima et al., "Development of a SiC Fibre with High Tensile Strength," Nature, Vol. 261, 5562, pp. 683-685, June 24, 1971.

6. Robert Riley and Terence Wallace, title unknown at present.

7. J. Economy, W.D. Smith, and R.Y. Lin, "High Strength Boron Carbide Fibers," Applied Polymer Symposium, No. 29, pp. 105-115, 1976.

8. J.O. Bird, C.W. Newquist, and E.L. Paquette, "On the Prediction of Properties of Advanced Fibrous Ceramic Composite Materials," to be presented at the Basic Science and Nuclear Division Joint Fall Meeting, American Ceramic Society, September 25-28, 1977, Hyannis, Massachusetts.

9. APPLICATIONS

A. DoD APPLICATIONS FOR CMCs*

Specific applications are recommended which have the potential for making a major impact. At temperatures less than 700°C, recommendations are:

1. Lightweight armor to defeat kinetic-energy penetrators
2. Antenna windows
3. Radomes

A possible armor configuration consisting of multiple layers of steel/ceramic/graphite-epoxy is suggested where the ceramic layers could be fiber-reinforced. Improved strength in such CMC layers could significantly improve the performance of the armor with respect to ability to fragmentate projectiles while sustaining minimum overall armor damage. The development of woven and impregnated SiO_2/SiO_2 and BN/BN composites is already well advanced and on the way to successful application in antenna windows. This is a good example of the successful reinforcement of a bulk ceramic material to improve mechanical strength and toughness to withstand moderate structural loads and temperatures. Improved radome materials are needed for ballistic missile interceptors to withstand high-G loadings due to rapid accelerations and maneuvering. Fiber-reinforced glasses are of great interest here and some consideration should also be given to fiber-reinforced ceramics such as silicon nitride.

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*Contributors: J.W. Warren and R.T. Pepper (who was not in attendance).

At temperatures greater than 700°C, recommendations are to concentrate on the high-temperature highly stressed turbine blades and vanes of small engines. Large sums of money have been expended on the development of ceramics, such as Si_3N_4 and SiC, aimed at increasing the operating temperature of turbines to the 2500°F regime. The available ceramic materials (Si_3N_4 and SiC) are inadequate on their own to meet the 2500°F goal. SiC has good creep strength but low toughness, and Si_3N_4 has good toughness but low creep strength. Composites combining fibers, such as SiC and a Si_3N_4 matrix, may have an excellent combination of strength and toughness and may be a solution to the turbine blade and vane problems.

Specific applications such as the above, which have high payoff potential, should be chosen as a motivation for fundamental and applied development programs aimed at demonstrating the technology of ceramic-matrix composites.

USES FOR COMPOSITE CERAMICS

MARKET / APPLICATION	PRODUCT	TEMPERATURE ≤800°C	TEMPERATURE >800°C
HEAT ENGINES			
OTTO	VALVES	X	
	VALVE SEATS	X	
	ROCKER ARMS	X	
	TAPPETS	X	
	PISTON CROWNS	X	X
	CYLINDER LINER	X	X
DIESEL	PRE CUP		X
	PISTON CROWN		X
	CYLINDER LINER		X
	CONNECTING ROD (2 CYCLE)	X	
	TURBO CHARGER ROTORS	X	
TURBINE	SHROUD	X	X
	COMBUSTOR		X
	STATOR VANES		X
	ROTOR		X
	TRANSITION	X	X
	SEAL	X	X
	MISC.	X	X
STIRLING	HEATER HEAD	X	X
FOSSIL FUEL/GEOTHERMAL			
	TURBINE COMPONENTS	X	X
	HEAT EXCHANGERS		X
	VALVES	X	X
	BEARINGS	X	X
	SEALS	X	X
PROCESS EQUIPMENT			
	HEAT EXCHANGERS		X
	PUMP COMPONENTS	X	X
	HEATING ELEMENTS		X
	REACTOR VESSELS	X	X
	PROCESS HARDWARE (FIXTURING)		X
	BEARINGS	X	X
	VALVES	X	X
	PISTON RINGS	X	X
	SEALS	X	X
	GAS IGNITERS		X
	MISC.	X	X

SPECIFIC D.O.D. APPLICATIONS WHERE C.M.C. MAY MAKE A MAJOR IMPACT

PHILOSOPHY FOR APPLICATIONS $> 700^{\circ}C$

- INITIAL DEVELOPMENT AND FABRICATION COSTS WILL BE HIGH AND APPLICATIONS HAVING CRITICAL PROBLEMS WITH HIGH PAYOFF POTENTIAL SHOULD BE SELECTED FOR TECHNOLOGY DEMONSTRATION.
- TO MINIMIZE COSTS DEVELOPMENT WORK SHOULD CONCENTRATE ON RELATIVELY SMALL ENGINES SUCH AS MAY BE USED ON THE CRUISE MISSILE.

SPECIFIC D.O.D. APPLICATIONS WHERE C.M.C. MAY MAKE A MAJOR IMPACT $> 700^{\circ}C$

HIGH TEMPERATURE HIGHLY STRESSED ROTATING PARTS SUCH AS TURBINE BLADES AND VANES.

PROBLEMS:

Si_3N_4 HAS GOOD TOUGHNESS BUT INADEQUATE CREEP STRENGTH.

SiC HAS GOOD CREEP STRENGTH BUT INADEQUATE TOUGHNESS.

SOLUTION:

REINFORCE Si_3N_4 WITH SiC FIBERS.

HIGH-PERFORMANCE C.M.C. MATERIALS

SYSTEMS ENGINEERING

THE SUCCESS OR FAILURE OF HIGH-PERFORMANCE COMPOSITE MATERIALS IN COMPETITION WITH CONVENTIONAL MATERIALS WILL NOT DEPEND UPON WHETHER THEIR PROPERTIES ARE SUPERIOR OR THEIR COST LOWER, BUT ON WHETHER THE FINAL PRODUCT MADE FROM THEM CAN OFFER BETTER VALUE THAN THAT ACHIEVABLE WITH THE MATERIALS NOW IN USE.

B. ADDITIONAL DISCUSSION OF APPLICATIONS FOR CMCs*

The most immediately obvious application for ceramic composites are as hot section components in advanced heat engines. As obvious as this application is, it should not be denigrated due to the payoff or return on investment potentially available; in fact, some of the returns are not immediately obvious. Helm of Detroit Diesel Allison and Kamo of Cummins Engine believe that the maintenance savings due to the elimination of the cooling system on ceramic diesels would justify the research effort, irrespective of efficiency gains. In addition to ceramic turbine components and Stirling-cycle heater-head tubes, many other applications exist, including:

- Heat Exchanger Tubes

  Particularly demanding application would be ceramic heat exchanger for a lightweight compact closed-cycle Brayton system on board naval vessels

- Mechanical Plasma Limiter Plates and First Wall Materials for Fusion Reactors
- Combustors

  MHD hot wall combustors
  Hot solid fuel dispersion devices

- Hot Gas Valve Components for MHD Regenerative Air Preheaters
- Plate Filters for Dirty Combustion Gases
- Hot Engine Seals and Submarine Main Shaft Seals

Weapons applications include:

- Nosetip and Leading-Edge Applications for Hypersonic Vehicles
- Radomes
- Armor Penetrators
- Gun Barrel Liners
- Beam Weapon Armor and Ceramic Armor
- Converging/Diverging Nozzles for Propulsion, Lasers, Fluid-Cooled Reentry Nosetips

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*Contributor: E.L. Paquette.

Industrial applications include:

- Ceramic Tools for In-Furnace Assembly Operations, Maintenance, etc.
- Unique Kiln Furniture
- Molten Corrosives Transport and Containment
- High-Duty Abrasives, All-Ceramic Tool Bits
- High-Temperature Friction, Wear, and Corrosion Applications
- Orthopedic-Match Modulus of Natural Bone and Provide Compatible Substrate for Calcium-Rich Glass/Bone Interface
- Waste Products--Production of glassy aluminum silicate fibers from coal-derived fly ash
- Primary Application as an Insulation Material (but also has potential to eliminate all-stone aggregate so that a very low-cost all-waste concrete can be developed)
- Structural Cement Vessels--including tanks, pipe, and castable boat or ship sections

SPECIFIC RETURN ON INVESTMENT: COMMERCIALIZATION

DATA:

2000 $MW_{THERMAL}$ OPEN CYCLE MHD BASELINE PLANT*

COPPER WATER WALL COOLING FLOWS:

3,377,303 LBS/HR WITH 50.5 BTU/LB ENTHALPY RISE.

CONCLUSION: COMBUSTOR COOLING RESULTS IN A 2.5% TOTAL SYSTEM ENERGY LOSS TO COOLING H_2O.

HOT WALL COMBUSTOR OPERATING AT 2400°F RATHER THAN 690°F WILL REDUCE ESTIMATED COOLING LOSS TO 1.3% OF TOTAL SYSTEM ENERGY INPUT.

THE 1.2% SAVINGS IS EQUIVALENT TO 26,000 TONS OF COAL PER ANNUM AT 100% OF DESIGN PLANT LOAD AND 80% AVAILABILITY.

PRESENT VALUE OF THESE SAVINGS OVER A 30 YEAR SYSTEM LIFE AT CURRENT PRICES OF $19.36/TON OF COAL IS $4,750,000 PER BASELINE PLANT.

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*ALL DATA ABSTRACTED FROM PAPER BY W.D. JACKSON, ET AL., "DEVELOPMENT OF A BASELINE REFERENCE DESIGN FOR AN OPEN CYCLE MHD POWER PLANT FOR COMMERCIAL SERVICE," 15TH SYMPOSIUM, ENGINEERING ASPECTS OF MHD, UNIVERSITY OF PENNSYLVANIA, MAY 24-26, 1976.

10. OVERVIEW OF SOVIET CERAMIC COMPOSITE TECHNOLOGY*

The Soviet Union is a leading country engaged in development and processing of engineering ceramic materials. Soviet interest in ceramic composite materials has avalanched in the past ten years as evident from the number of publications and patents. The current emphasis does not appear to be decreasing. Analysis of their research suggests that the Soviets have an extensive developmental program under way to synthesize advanced composite materials based on ceramics (Fig. 1). Synthesis of both ceramic whiskers and filaments has been noted in addition to their incorporation into ceramic matrix composites. Such a program would be based on a wide range of inorganic fibrous materials with extraordinary properties and on an increased understanding of the mechanics of brittle matrix materials. The USSR possesses a well-developed basic technical capability to utilize engineering ceramic materials for unique structural applications required by the military: optical and microwave windows, missile radomes and nose cones, lightweight structural engineering members, bearings, gas turbine components, and protective and thermal control coatings.

At least 19 institutes have been identified as conducting research on ceramic matrix and fiber materials (see Fig. 2 and Table I following the figures). Since the early 1960s, the Soviets have initiated various R&D programs involving ceramic fibers/whiskers and ceramic-matrix composite materials (Figs. 3-13). The work has significantly increased since about 1970

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*Contributors: W.J. Bover and C.A. Petschke (who was not in attendance).

as judged by a review of their scientific literature. For example, at least 51 Soviet author certificates have been publicly released between 1970 and 1976 (Fig. 14). Before 1970, ceramic composite work was reported relatively infrequently.

The Soviets clearly recognize the advantages of strengthening monolithic materials by combination with fibers. The primary advantage is the use of the resulting composite at higher temperatures than the matrix alone (Figs. 15, 16). Also, the Soviets clearly recognize that much work is required if fiber-reinforced glasses and ceramics are ever to become acceptable replacements and viable design alternatives to more conventional metallic materials. The Soviets are also active in the development of carbon-carbon composites (Fig. 17).

The overall evidence indicates that the Soviets are strong in theoretical work and weak in practical applications. The resources allocated to the field of ceramic composites are impressive, but the overall results have been disappointing, at least from the standpoint of developing new and practical applications. A significant amount of what the Soviets have published tends to be repetitive and perhaps can best be described as derivative rather than original. In other words, analysis suggests that roughly a quarter of their effort appears to be devoted to reviewing the work of others. Nevertheless, the Soviets are bringing a large amount of resources to bear on developing ceramic-matrix composite materials. The precise extent to which they have been able to make these materials and use them remains unknown. However, no specific defense-related connection has been established, yet, for the Soviet work.

The translation of whisker properties into composites has not been successful worldwide, primarily due to fabrication difficulties such as sorting, alignment or dispersion, and quality control problems. It is believed that short ceramic

fibers or whiskers probably would meet Soviet needs of moderate-strength but low-cost composites for complex shapes. These materials are not in direct competition with modern filamentary composites such as those employing boron, silicon carbide, or graphite fibers. Soviet capability exists for synthesis of whisker-reinforced ceramic-matrix composites (Figs. 14, 15, 18); however, large-scale production of reliable structural components to meet design needs is questionable because of general Soviet deficiencies in fabrication know-how.

In order to effectively apply brittle ceramics as critical load-bearing components, sophisticated design methods must be developed. Recent Western work has been exploring the use of fracture mechanics in evaluating and designing brittle structures. It has only been in the past few years that the Soviets have reported on modern fracture mechanics theories of brittle materials components designed to withstand mechanical and thermal stresses. Analysis of Soviet publications indicates that they are closely monitoring Western programs on brittle materials design. It is likely that in recent years the Soviets have extended their composite fracture mechanics research to include the study of ceramic composite material.

It is evident that the Soviets are active in the development of high-performance ceramic materials (Fig. 19). There is no indication, however, that the Soviets have advanced out of the basic or exploratory stages of the development of these materials.

FIGURE 1

SOVIET CERAMIC FIBERS

RECOGNIZE STRENGTH RETENTION AT HIGH TEMPERATURE

R&D ACTIVITY - POLYCRYSTALLINE FILAMENT

- MULTIPHASE FILAMENT
- MONOCRYSTALS (WHISKERS)

GOALS SET IN 1976-80 FIVE-YEAR PLAN

FIGURE 2

INSTITUTE	REINFORCEMENT	
	WHISKER	FIBER
INSTITUTE OF PROBLEMS IN MATERIAL SCIENCE	MULLITE ZrO_2	SIC
BAYKOV INST OF METALLURGY	-	SIC, Al_2O_3
INST OF AVIATION MATERIALS	ALN	SIC, Al_2O_3
VORONEZH STATE UNIV.	SIC, Al_2O_3	-
MOSCOW CHEM-TECH INST	BEO, MGO, SIC ALN	-
INST OF CRYSTALLOGRAPHY	ALN, TiO_2	-
LABORATORY OF BASALT FIBERS, KIEV	-	BASALT

FIGURE 3

ALUMINUM OXIDE

SAPPHIRE WHISKERS - EARLY 1960S

ALPHA-AL_2O_3 FILAMENTARY CRYSTALS - 1969

CONTINUOUS MONOCRYSTALLINE AL_2O_3 FILAMENTS IN TI MATRIX - 1972

AL_2O_3 FIBER EXTRUSION PROCESS LESS EXPENSIVE THAN US PROCESS

FIGURE 4

MULLITE SINGLE CRYSTALS

DEVELOPMENT PRIOR TO 1970

USED WITH METALLIC AND CERAMIC MATRICES

FURTHER DEVELOPMENT OF MULLITE EXPECTED

FIGURE 5

SILICON CARBIDE

1964 BETA - SIC WHISKERS

1968 SURVEY ARTICLES ON SIC FIBERS

COMPATIBILITY STUDIES OF SIC WITH W, TI, EPOXY

DEVELOPMENT OF COMPATABLE BARRIERS FOR SIC IN METAL MATRIX

1975 FORMATION OF SIC VIA LASER HEATING

FIGURE 6

SILICON CARBIDE PRODUCTION

PRODUCTION CONDITIONS IN OPEN LITERATURE

TENSILE STRENGTHS 690 MPA < US VALUES

SOVIET INTEREST IN PLASMA FORMATION OF MATRIX WITH SIC FIBER

SOVIET SIC WORK FOLLOWS US LEAD

FIGURE 7

ZIRCONIUM DIOXIDE

ACICULAR ZrO_2 MONOCRYSTALS IN ZrO_2

INCREASED THERMAL SHOCK RESISTANCE

FIGURE 8

ALUMINUM NITRIDE

1970 ACTIVE WHISKER R&D PROGRAM

1971 USE IN POLYMERIC MATRIX

1973 ESTABLISHMENT OF NEW PRODUCTION FACILITY

AlN DEVELOPED FOR USE IN COMPOSITES

FIGURE 9

MAGNESIUM OXIDE

R&D SINCE MID-1960s

MgO WHISKERS IN MgO COMPOSITES IN 1970

OPTIMIZATION OF PRODUCTION TECHNIQUES IN 1974

NO SOVIET PATENTS FOR THESE WHISKERS

FIGURE 10

MINERAL FIBERS

CONSIDERABLE R&D ACTIVITY IN COMPLEX MIXED OXIDE TECHNOLOGY

USE OF SLAG WOOL STAPLE FIBERS IN SOVIET COMPOSITES

BASALT STAPLE FIBER PRODUCTION IN MID-1960S

SUPERFINE (0.5 μM) BASALT PRODUCTION TECHNOLOGY IN MID-1970S

BASALT FIBERS USED AS CONSTRUCTION MATERIAL AND REINFORCEMENT IN PLASTICS

SOVIET CORPORATION FORMED TO TRANSFER TECHNOLOGY FROM R&D INTO INDUSTRY

FIGURE 11

SYNTHETIC ASBESTOS

EFFORTS BEGAN IN EARLY 1960S

SYNTHESIS OF FLUOROAMPHIBOLES

POSSIBLE PRODUCTION OF STABLE FIBERS FOR SPINNING, 1972

FIGURE 12

TYPICAL REPORTED FIBER PROPERTIES

CERAMIC MATERIAL	DENSITY g/cm^3	TENSILE STRENGTH GPa	YOUNG'S MODULUS GPa	DIAMETER μm	LENGTH mm	DOI
α-Al_2O_3	3.9	5.6	441	3-60	-	1975
MULLITE	3.1	1.7	196	0.3-5	0.15-10	1973
ZrO_2	5.56	0.29	-	5-7	0.15-0.25	1974
SiC	-	2.0	480	100	FIBER	1973
AlN	-	6.9	320	5-10	4-20	1970-74
MgO	-	17	-	1-3	30	1976

FIGURE 13

DIFFUSION BARRIERS

SiC, ZiC, TiN, AND ZrN ON CARBON FIBERS FOR Al MATRIX

TiN, ZrC, Al_2O_3, SiO_2, AND BN COATINGS ON W, Mo WIRE

TiN COATING ON W FOR USE IN Ni MATRIX

FIGURE 14

SOVIET CERTIFICATES PUBLICLY RELEASED BETWEEN 1970-1976

YEAR ISSUED	CERTIFICATE NO.	WHISKER	VOL % WHISKER	MATRIX
DEC 1972	342845	KAOLIN	0.5 - 4 %	AL_2O_3 AND KAOLIN
OCT 1974	494372	CARBON	15-20	AL_2O_3
AUG 1973	395342	MULLITE	12-20	AL_2O_3 AND MO
JUL 1973	392050	MULLITE	6-12	$MoSi_2$
JUL 1973	390051	SIC	85-90	Si_3N_4
AUG 1975	481578	MGO	13-20	ZrO_2
MAR 1974	420599	MULLITE	10-20	ALN AND MO

FIGURE 15

MECHANICAL PROPERTIES OF CERAMIC COMPOSITES

MATRIX	VOL % OF α AL_2O_3	IMPACT STRENGTH $KG\cdot CM/CM^2$	BENDING STRENGTH MPA	COMPRESSIVE STRENGTH MPA	NO OF CYCLES 1200°C TO 20°C TO FAILURE
MgO	0	2.1	80	-	1
MgO	15	6.5	240	-	50
ZrO_2	0	3.5	108	800	10
ZrO_2	15	7.2	220	1200	50
BN	0	3.4	52	-	5
BN	15	8.5	98	-	30
Si_3N_4	0	1.2	55	250	20
Si_3N_4	15	22.0	310	720	100

FIGURE 16

ZrO_2 WHISKERS IN ZrO_2

% ACICULAR ZrO_2 WHISKERS	% OPEN POROSITY	ULTIMATE BEND STRENGTH, MPa	IMPACT STRENGTH $KG \cdot CM/CM^2$	NO. CYCLES IN AIR FROM 1300°C TO 20°C BEFORE FRACTURE
0	0.9	127	2.4	22
5	1.2	147	4.0	50
10	1.4	166	4.5	100
15	1.8	149	6.0	100
20	3.8	145	5.8	100

FIGURE 17

CARBON/CARBON

1970 - PATENT FOR C/C COMPOSITE

1972 - C/C SUGGESTED FOR USE UP TO 2000°C

1975 - C/C AVIATION BRAKES

FIGURE 18

MULLITE WHISKERS IN CERAMIC MATRICIES

MATRIX MATERIAL	VOL % MULLITE	COMPRESSION STRENGTH MPA	BENDING STRENGTH MPA	IMPACT STRENGTH $(KG \cdot M)/CM^2$	NO. CYCLES IN AIR FROM 1000°C TO 20°C BEFORE FRACTURE	MAXIMUM USABLE TEMP °C
Al_2O_3	?	1379	284	8.5	150	2000
ZrO_2	15	1179	241.6	8.5	150	2000
Cr_2O_3	?	637	156.5	21.1	100	1900
TiO_2	?	274.4	117.9	6.0	50	1900
AlN	20	343.4	99.9	16.0	500	1900
Si_3N_4	20	900	-	20.0	400	-
BN	?	678	-	49	746	-

FIGURE 19

SUMMARY

ACTIVE R&D PROGRAM

SOME CERAMIC FIBERS AND WHISKERS ARE PRODUCED ON COMMERCIAL SCALE

EXTENT OF USE OF CERAMICS IS UNKNOWN

COMPOSITE FABRICATION TECHNOLOGY CAPABILITY IS QUESTIONABLE

SOVIETS APPEAR TO HAVE GENUINE INTEREST IN DEVELOPMENT OF STRUCTURAL CERAMIC COMPOSITES

TABLE I. PERSONALITY/FACILITY LOCATION LIST

Organization and Location	Names	Field of Study
Institute of the Problems of Materials Science (IPROMAT), Kiev	Frantsevich, I.N. Listovnichaya, S.P. Karpinos, D.M. Panasevich, V.M. Panasyuk, A.D. Tresvyatskii, S.G. Zil'berberg, V.G. Samoylenko, V.G. Grosheva, V.M. Gayovoy, T.I. Volkogon, L.M. Vishnevskii, V.B. Artemov, V.A. Pavlikov, V.N. Mikhashchuk, Ye. P. Fedorenko, V.K.	Mullite whiskers in oxide matrices. SiC fibers in Ti, Al. Barrier coatings. ZrO_2 whiskers. Al_2O_3 platelets.
Institute of Metallurgy imeni Baykov, Moscow	Shorshorov, M. Kh. Kudinov, V.V. Antipov, V.I. Katinova, L.V.	SiC fibers in Al, Ti. Barrier coatings. Al_2O_3 fibers.
Moscow State University, Moscow	Sokolovskaya, Ye. M. Tsukerman, S.A. Kravchenko, A.T. Ivanova, V.S. Botvina, L.R.	SiC fiber in Ti.
All Union Scientific Research Institute of Aviation Materials (VIAM), Moscow	Shakov, Yu. A. Tumanov, A.T. Portnoy, K.I. Gunyaev, G.M. Gribkov, V.N. Isaykin, A.S. Bokshtein, S.Z. Kishkin, S.T. Silaev, V.A. Levinskii, Yu. V. Umantsev, E.L. Moldavskii, V.M. Shchetanov, B.V. Bel'mer, P.F. Zaitsev, G.N. Nazarova, M.P. Svetlov, I.L. Sorina, T.G. Salibekov, S.E. Dvoichenkova, L.V. Trefilov, B.F.	SiC,fibers in Ti. Al_2O_3 filaments. AlN whiskers. Barrier coatings.
Titanium Institute, Moscow	Anohin, V.M.	Ti strengthened with TiO_2, ZrO_2, carbides, nitrides, silicides.

Table I. Cont.

Organization and Location	Names	Field of Study
State Scientific Research Institute of Machine Studies, Moscow	Lyuttsau, V.G. Stepanichev, Ye. I.	TiO_2 coatings.
Tomsk University, Tomsk	Kashin, O.A. Zayats, I.I. Bakach, G.P. Borisov, M.D.	Barrier coatings.
Voronezh State University, Voronezh	Drozhzhin, A.I. Belikov, A.M. Kosilov. A.T. Belyaev, A.M. Yeresnev, M.A. Shulik, B.V. Yuyukin, K.P. Grozdev, A.D. Fostnikov, V.S. Karelin, B.V. Moskalenko, A.G. Petrov. V.N. Shchetinin, A.A. Kutakov, K.S. Postnikov, V.S. Ammer, S.A.	SiC, Alumina whiskers
Moscow Chemical-Technological Institute imeni D.I. Mendeleyev, Moscow	Vlasov, A.S. Gavrik, G.G. Zakharov, Ye. K. Spirina, T.V. Metushevskii, A.S. Timashev, V.V. Kudryashov, V.V. Kotlyarova, T.V. Rasskazova, L.A. Slinkin, Yu. I. Pichugin, Ye. F. Karakanidi, N.G. Kibardin, R.N. Melkumyan, E.S. Khozhainov, Yu. M. Karpenko, A.D. Budnikov, P.P. Sandulov, D.B.	Whiskers of BeO, MgO, MoO_3, SiC, and AlN.
Institute of Casting Problems, Kiev	Karpinos, D.M. Mikhashchuk, Ye. P. Efimov, G.V. Pavlikov, V.N. Artemov, V.A. Efimov, V.A.	Al_2O_3 flake crystals

Table I. (Cont.)

Institute of the Problems of Strength, Kiev	Gogotsi, G.A. Grushevskii, Ya. L. Kurashevskii, A.A. Artemov, V.A. Gashchenko, A.G. Rudak, I.N. Strelov, K.K. Pisarenko, G.S. Karaulov, A.G.	Structural mechanics.
Scientific Laboratory of Basalt Fibers, Kiev	Ambrosivenko, V.V. Makhova, M.F. Alekseyev, A.I. Guzhavin, O.V. Gorodetskaya, S.V.	Basalt fibers.
Institute of Silicate Chemistry imeni I.V. Grebenshchekov, Leningrad	Fedoseev, A.D. Grigor'eva, L.F. Makarova, T.A. Korytkova, E.N. Nesterchuk, N.I. Krupenikova, Z.V.	Synthetic asbestos. Barrier coatings.
Urals Polytechnic Institute imeni Kirov, Sverdlovsk	Mamykin, P.S. Kashcheev, I.D. Perepelitsyn, V.A.	MgO whiskers.
Institute of Geology and Geophysics (Siberian Branch), Novosibirsk	Kalinin, D.V. Lokhova, G.G. Deniskina, N.D. Shurupov, Yu. V. Shvedenkov, G. Yu.	Synthetic asbestos.
Institute of Crystallography, Moscow	Golosova, M.A. Papkov, V.S. Berezhkova, G.V. Rozhanskiy, V.N. Zakharov, I.N. Koryukin, V.I.	Whiskers of AlN, TiO_2, Al_2O_3.
Institute of Metallurgy, Academy of Sciences, Georgian SSR, Tbilisi	Tavadze, F.N. Kiyanenko, V.V. Makovets, S.E. Surmava, G.M. Nikolaishvili, A.A.	SiC whiskers.
Institute of Applied Chemistry, Leningrad	Fridlender, B.A. Neshpor, V.S. Ermakov, B.G. Sokolov, V.V.	TiN whiskers.
Institute of High Temperature, Moscow	Samsonov, V.P. Rautbort, A.E. Val'yano, G.E. Serebrennikova, V.E. Prokhorova, I.V.	SiC whiskers

11. OUTLINE OF NOTES ON WORKSHOP

A. MATERIALS SYSTEMS

For atmospheric temperatures, perhaps up to 300°C, and quite high temperatures in a nonoxidizing environment.

Oxide/refractory metal, fibre/oxide matrix composite, made by directional solidification of eutectic. Fibres to be kept smaller than the critical flaw size of the matrix.

Examples: W/ZrO_2 stabilized with Y_2O_3. Fibres are single crystals, 0.2-1.0 μm in diameter. $MoGd_2O_3$ doped with CeO_2, made as above, crushed, then hot-pressed. (Strength 1-2 orders of magnitude greater than that of the oxide alone.) [Chapman, Bradt]

For atmospheric temperatures.

Fill porous ceramic with polymer, for double or triple strength.

For temperatures upward of 1400°C.

SiC/Si Milled SiC plus thermosetting plastic, injection-molded, reaction-sintered with Si provided by SiF_4 (or liquid Si?) [Whalen]

[On 14 September 1977, Whalen was asked about the feasibility of adding carbon in the final stage (to use up the excess Si) in the manner of the gas-carburizing of steel. He replied, "We're trying that now." He thought, however, that because the SiC/Si body is "fully dense," the carburizing would be confined to the surface.]

For temperatures to 2500°C, or higher.

C/46 vol pct NbC	Hot-pressed at 2850°C or more for chemical bonding.
C/TaC	Graphite woven cloth plus Ta from $TaCl_4$. Makes isotropic body. [Riley]

Service temperature is limited by eutectic of each carbide-carbon system.

For temperatures up to 1400°C, perhaps higher.

Si_3N_4/Ta fibres 0.025 inch	Hot-pressed. 30-fold increase in impact strength but oxidation resistance less than that of Si_3N_4. [Brennan]

For temperatures up to about 600°C.

7740 Glass/0.056" Borsic or 0.057 Borsic	(Other glasses might increase range above 800°C.) Other reinforcements of some promise: SiC, Graphite, Al_2O_3 FP(duPont) [Prewo]

For unspecified temperature (but high).

Graphite/BN reinforcement [Paquette]

General concept for tough CMC: Depends largely upon energy absorption by:

- Toughness of the reinforcing fibre itself
- Pull-out of fibres
- Phase transformation
- Line tension (interparticle spacing must be small with respect to length of crack.)
- Microcracking
- Dispersion strengthening [Rice]

Other high-temperature systems.

Al_2O_3/30 vol pct BN.	Very rough fracture surface indicates toughness.	
B_4C/50 Graphite.	Same observation.	[Rice]

B. MODES OF FABRICATION

Make fibre-reinforced composite by unidirectional solidification of eutectic; crush; hot-press. Great improvement in strength. [Chapman]

Can make either fibrous or lamellar reinforcement by directional solidification.

Spacing between lamellae or fibres is linearly, but inversely proportional to the solidification rate.

Crystallography of the oxide matrix is wholly determined by the oxygen sublattice.

An oxide/oxide composite is best built *in situ*.

A strong interfacial bond is required for service above 1000°C. [Bradt]

Make SiC/Si composites (some are already commercial) thus:

- Mill the SiC to size
- Mix with thermosetting plastic
- Injection-mold
- Add Si (is it liquid Si or SiF_4?)
- Reaction-sinter
  - Shrinkage during carbonizing of plastic; none in siliciding.
  - Leaves 8-10 percent of free Si.
  - Now trying to add C, finally, to react with excess Si. [Whalen]

In the hot-pressing of carbide/carbon composites, a chemical bond is developed only if the temperature is 2850°C or higher.

In new method at LASL:

> Woven carbon cloth is supplied Ta from $TaCl_4$. The Ta coats all fibres. Microstructure proves the body to be highly isotropic. [Riley]

In preparation of assembled macrofibre-reinforced CMC, a planned uneven distribution of the fibres (e.g., more dense in the core than near the surface of a body) is entirely feasible.

Compaction by explosives has had some attention (AlN, BeO, Si_3N_4, Al_2O_3, others). [Cline]

Rough treatment during manufacture may greatly strengthen a ceramic.

> Phase-transformation toughening may occur in machining [by compressive stress (perhaps torsional too) applied by the tool]. For example, partially stabilized ZrO_2.

Copyrolysis promotes uniformity, strengthening (2-3 orders of magnitude) of glassy carbon.

Sol/Gel methods produce homogeneous composites of ZrO_2 in Al_2O_3. (But sometimes a bit of heterogeneity is desirable.) [Rice]

C. INNOVATIONS

Roy Rice thinks to splat-cool ceramic eutectics to achieve uniform microstructure, then to collect the material and process it in conventional modes.

Carl Cline sees advantage in alloy development for metallic fibres, thinking especially of alloys of W, of Be.

Cline expects value in oriented microstructures. He foresees that they could reaily be built in Al_2O_3.

Cline thinks that we should develop the potential of high pressures in ceramic processing.

Paquette reported that ribbons of graphite have been made, only about $2x10^{-4}$ inch thick.

Paquette cited progress in pressure slip casting of Al_2O_3/Al_2O_3 by Condon and Lewis at Alfred University (maybe that is the way to make radomes).

12. LIST OF WORKSHOP ATTENDEES

Arden L. Bement, Jr.	DARPA
William J. Bover	Army Foreign Science & Technology Center
Richard C. Bradt	Penn State University
John J. Brennan	United Technology Research Center
A.T. Chapman	Georgia Tech
Carl F. Cline	Lawrence Livermore Laboratory
H.M. (George) Davis	Consultant, IDA
Winston Duckworth	Battelle Columbus Laboratories
Herbert Herman (not present)	SUNY-Stony Brook
John E. Hove	Institute for Defense Analyses
Robert A. Lad	NASA-Lewis Research Center
Edward L. Paquette	Atlantic Research Corp.
Roger T. Pepper (not present)	Fiber Materials, Inc.
Karl M. Prewo	United Technology Research Center
Roy W. Rice	Naval Research Laboratory
Robert E. Riley	Los Alamos Scientific Laboratory
James W. Warren	Warren Associates
Thomas J. Whalen	Ford Motor Company

APPENDIX B

RESPONDENTS TO IDA SURVEY

Iqbal Ahmad	Benet Weapons Labs-Watervliet Arsenal
Richard Alliegro	Norton Company
R.L. Ashbrook	NASA-Lewis Research Center
A.N. Baker	Lockheed-California Company
Robert Beasley	Lockheed Missiles and Space Company
Warren Beck	3M Company
Charles F. Bersch	Naval Air Systems Command
Jean Blachere	University of Pittsburgh
Seymour Bortz	IIT Research Institute
S.W. Bradstreet	Consultant
Richard Bradt	Pennsylvania State University
John Brennan	United Technologies Research Center
Sherman Brown	University of Illinois
J.E. Burke	General Electric Company
Harry W. Carpenter	Rocketdyne
A.C.D. Chaklader	The University of British Columbia
A.T. Chapman	Georgia Institute of Technology
Gordon P.K. Chu	GTE Sylvania Incorporated
Carl Cline	Lawrence Livermore Laboratory
Thomas Courtney	Michigan Technological University
Ivan Cutter	University of Utah
Keith Davidson	University of California
R.C. DeVries	General Electric R&D Center
Arthur Diness	Office of Naval Research
Robert Doremus	Rensselaer Polytechnic Institute
Winston Duckworth	Battelle Columbus Laboratories
Jules Duga	Battelle Columbus Laboratories
Sunil Dutta	NASA-Lewis Research Center
Pol Duwez	California Institute of Technology

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Morris Fine	Northwestern University
Henry Graham	AFML/LLM Wright-Patterson Air Force
Yoshiro Harada	IIT Research Institute
Herbert Herman	State University of New York-Stony Brook
William Hillig	General Electric R&D Center
Jerome Krochmal	Wright-Patterson Air Force Base
Henry P. Kirchner	Ceramic Finishing Company
David Kupperman	Argonne National Laboratory
Charles Lynch	Wright-Patterson Air Force Base
Irving Machlin	Naval Air Systems Command
Norman Macmillan	Pennsylvania State University
James McCauley	Army Materials & Mechanics Research Center
Thomas McGee	Iowa State University
A.F. McLean	Ford Motor Company
Mel Mendelson	Pratt & Whitney Aircraft Group
John Milewski	Exxon Research & Engineering Company
P.E.D. Morgan	University of Pittsburgh
James Mueller	University of Washington
Fred Ordway	Artech Corporation
Edward Paquette	Atlantic Research Corporation
Roger Pepper	Fiber Materials, Inc.
Bert Phillips	LeMont Scientific, Inc.
S.R. Pollack	University of Pennsylvania
Karl Prewo	United Technologies Research Center
R.W. Rice	Naval Research Laboratory
M.H. Richman	Brown University
Ralston Russell	Ohio State University
Hroshi Sato	Purdue University
Alan Searcy	University of California
A.C. Siefert	Owens-Corning Fiberglas Corporation
George Sines	University of California

William Smothers	Bethlehem Steel Corporation
Harold T. Smyth	Rutgers University
M.A. Steinberg	Lockheed Aircraft Corporation
Stephen D. Stoddard	University of California
Norman M. Tallan	Wright-Patterson Air Force Base
James Tinklepaugh	New York State College-Alfred University
Minorn Tomozawa	Rensselaer Polytechnic Institute
Richard Tressler	Pennsylvania State University
Joseph Uher	Pemco Products Group
Stephen Urban	Transtechnovations, Inc.
E.C. VanReuth	Defense Advanced Research Projects Agency
Dennis J. Viechnicki	Army Materials & Mechanics Research Center
John Wachtman	National Bureau of Standards
Franklin F.Y. Wang	State University of New York-Stony Brook
James Warren	Warren Associates
Thomas Whalen	Ford Motor Company
Gerald Wirtz	University of Illinois

APPENDIX C

TECHNICAL SURVEY QUESTIONNAIRE ON CERAMIC-MATRIX COMPOSITES

IDA
INSTITUTE FOR DEFENSE ANALYSES

SCIENCE AND TECHNOLOGY DIVISION
400 Army-Navy Drive, Arlington, Virginia 22202 • Telephone (703) 558-1000

Dear Colleague:

As part of conducting an on-going study of the technological programs of the Department of Defense involving materials and structures (a study being done by IDA for the Office of the Director of Defense Research and Engineering), we are exploring the potential of advanced Ceramic-Matrix Composites for structural use. Our aims are (1) to identify possible advantageous applications, (2) to evaluate the current state of underlying technology, and (3) to establish guidelines for any future activity of the DoD in this field. For the moment, the meaning of "Ceramic-Matrix Composites" may be broadly interpreted, the two restrictions being that the concept excludes assembled macrostructures and that carbon-carbon composites, per se, will not be considered.

Your opinions and comments are earnestly solicited, and will be gratefully received. As an indication of the sort of information desired, a brief questionnaire of simple format is enclosed, but you should feel free to organize your reply in another fashion if you prefer.

Like all who are asked to participate in this survey, you have been selected on the basis of your experience and professional standing. Your response to the enclosed questions therefore constitutes a valuable contribution to the planning of future DoD technology base programs involving such composites.

If at all possible, a reply within two weeks would be highly desirable.

Sincerely,

John E Hove

John E. Hove

H M Davis ("George")

H.M. (George) Davis

Encl.

IDA SURVEY ON CERAMIC-MATRIX COMPOSITES

Name ______________________________

Mailing Address ______________________________

____

______________________________ Zip __________

Business Telephone ______________ Area Code __________

Background (approx yrs.)	R&D	Manufacture	Use
–in Ceramics	___	___	___
–in Composites	___	___	___
–in Ceramic-Matrix Composites	___	___	___

1. Do you know of, or can you conceive, structural functions in which Ceramic-Matrix Composites might provide superior service? Yes___ No___

If you answered "yes", will you please cite examples? Any discussion you care to add would be welcome.

2. In your judgement, is the development of useful structural Ceramic-Matrix Composites

a) feasible ___,
b) a difficult possibility ___, or
c) probably impossible ___?

3. Given the problem of (2), suggest systems that you consider worthy of investigation toward the development of structural Ceramic-Matrix Composites. Please discuss briefly.

4. Other comments or suggestions.